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## A THEORY OF COLLOID BEHAVIOR IN DOUGH

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In writing this paper the author has sought to gather together as many recent ideas related to this subject as were feasible. It would have made the paper unduly long to compose it in the conventional style in which special mention is made of different authors and their publications. A blanket acknowledgment is hereby made to all from whose work ideas have been taken. The only originality claimed by the author is the fusing together of these different ideas.

Dough forms one of the most complex colloid systems. It is composed of different substances and the quality of the dough and the resulting bread depends on the nature of these substances, their relationship and interactions among each other. It is the purpose of this paper to present a brief word picture of this colloid system.

### Proximate Analysis of Flour

Proximate analysis of wheat flour shows that it contains protein, which includes all the nitrogenous compounds; ash, composed of a number of different elements; carbohydrates, mostly starch, a little sugar and cellulose; fat, and water. The nitrogenous material includes the several individual proteins: gliadin, glutenin, leucosin, edestin, and proteose. Besides all these there are present enzymes, principally amylases and proteases. The former influence the rate of starch hydrolysis; the latter, the rate of proteolysis which may be harmful or beneficial depending on conditions. Just how the elements which make up the ash or mineral matter are combined is not known. We know that a certain amount will enter the water solution phase. Besides all these are the lipoids principally phosphatides, important because of their influence on colloid phase relations.

When dough is made by mixing water, flour, yeast, shortening, salt, and sugar, we have a most complex colloid system. In order to understand the factors which determine the quality of dough and resulting bread it is necessary to look at this system as a whole.

### The Particle as the Unit of Colloids

The substances which compose the dough exist in the colloid condition, and the properties imposed by this condition determine its quality and that of the resulting bread. In the same way as the molecule is used in general chemistry as a center around which to organize ideas, the particle may be said to be the organizing center of colloid chemistry. The colloid particle may be considered as an aggregation of smaller molecules or perhaps a very large molecule. Any way we deal with a unit much larger than the molecule of an ordinary solution. These colloid particles may be formed by the disintegration of larger masses into submicroscopic or ultra microscopic units, or they may be formed by the aggregating of diffused smaller molecular units into larger units which we call particles. The smallest flour grain is a mass of colloid particles.

#### Characteristics of the Particle

The size, shape, internal and external form—in other words, the structure of the particle—has a determining effect on the properties of the colloid. One class of particles may be spherical, cubical, or irregular in shape, but no matter what the external form, with them are associated the idea of a solid or internal homogeneity. Colloid clay would be an example of this class. Another class of particles are made of aggregates of smaller units or molecules associated in such a way that we do not think of them as possessing a homogeneous structure. Such particles are considered to have a mesh or network structure. Substances present in flour belong more to this class than to the other. In this class of particles there are many variations in structure: The protein particles behave as tho they had a more pronounced mesh or network structure than the starch particles. By protein particles we mean associations or aggregations of nitrogenous material which act as a unit. Whether these units exist in the flour as aggregates of different proteins, such as gliadin and glutenin, or whether these proteins unite as soon as water is added is immaterial. We know that as soon as flour and water are mixed, we have the particles of which gluten is made.

The terms sol, hydrosol, gel, and hydrogel are used in connection with colloid systems. A sol or hydrosol is a colloid solution in which water is the disperse medium. A gel results when the particles in a sol are massed in an aggregate. From this standpoint gluten is a gel.

#### Colloid System Is Polyphase

Substances in the colloid state form polyphase systems. With such systems are associated the ideas of surfaces and interfaces. The



relation of surfaces to each other at interfaces and the extent of total surface determine some of the fundamental properties of substances in the colloid state. The amount of total surface is directly related to the size of the particles and to the configuration of the surface. A spherical surface is of the smallest extent in relation to mass. The more irregular the surface, the greater the extent of surface in relation to mass. The greatest extent of surface is found in particles having a mesh or network structure. Protein particles of flour behave as tho they had a much greater amount of surface in proportion to mass than starch particles. Hence we think of protein particles as having a network, or wadded mesh structure. This great extent of surface is associated with some of the fundamental properties of the protein particles which, when united in strands, chains, or aggregate, form the gluten.

### Phase Relations in Colloids

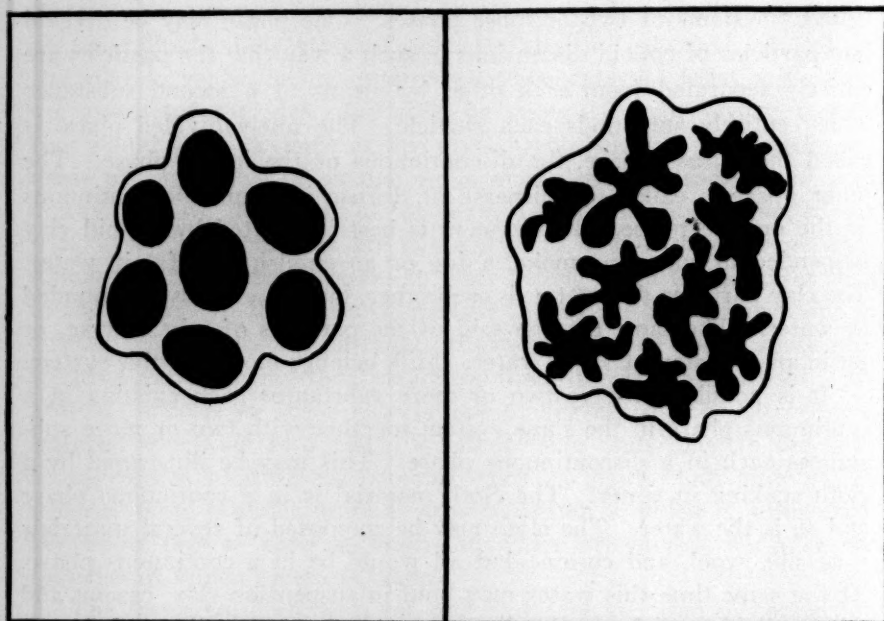
From what has been said it follows that colloids form heterogeneous systems of two or more phases. One phase may be divided into particles of colloid dimensions in such a way that the particles are entirely separated from each other by means of a second substance which entirely surrounds each particle. The finely divided phase is called the diffuse phase, the discontinuous or the inside phase. The other phase is called the disperse or diffuse medium, the continuous or the outside phase. This system is best illustrated by colloid clay suspended in water, a smoke, a fog or an emulsion of fat in water. The clay particles do not touch each other, but are entirely surrounded by water. The same may be said of the particles of mist, smoke, or fat in an emulsion of fat in water. Milk belongs mostly to this system.

It is possible to have two or more substances each existing in a continuous phase in the same system together with two or more substances each in a discontinuous phase. This may be illustrated by a cloth soaking in water. The cloth material is in a continuous phase and so is the water. The cloth may be composed of several materials—as silk, wool, and cotton—but all would be in a continuous phase. At the same time this water may hold in suspension clay, casein, and other colloid particles in the discontinuous phase as well as substances in true solution. This gives us some idea of the possible complexity of colloid systems.

### Dough as a Colloid System

The most apparent physical property which makes it possible to form dough by mixing a certain amount of flour with water is that

water wets the flour or flour adsorbs water. The wheat flour grains may be considered as being built of a large number of colloid particles, principally starch and proteins. When water is added to flour the grains are enveloped in a film of water, and the water also penetrates the grains and each colloid particle is surrounded by water films each in contact with its neighbors. The water thus forms a continuous phase. If enough water is added to more than fill the voids among the flour grains as well as interstitial spaces, the flour grains disintegrate and no dough is formed. We know that if we mix flour and water, say 1 to 10, we do not get a dough but a suspension. If this suspension is centrifuged, and the supernatant liquid is poured off we obtain a dough. To form dough it appears necessary that the particles approach each other close enough to make some kind of contacts. We know that the stiffness of a dough is intimately related to the amount of water present or to the thickness of the film of water which surrounds the colloid flour particles.



Starch And Water

Protein And Water

FIGURE 1

Figure 1 illustrates a two-phase system: starch and water or protein and water. The water forms the continuous phase, while the starch or the protein form the discontinuous phase. The forces inherent in a water film account in a large part for the coherence among the starch particles. This coherence would be strengthened by increase of surface such as may be imagined in the case of the complex surface of the protein particles.

The colloid starch particles and the colloid protein particles differ in internal structure. It is probable that the water wets only the surface of the starch particles in dough, which means that the starch particles behave more as a solid. Penetration of water into the interior of the starch particles evidently takes place when starch is hydrolyzed. The internal structure of the protein colloid particles is such that water not only wets the surface but penetrates into the interior or the protein particles behave more as though the structure were a mesh similar to a sponge or a wad of cotton. When water is added to this protein particle, it not only covers the outside but penetrates into the interior and each strand is covered with a film of water. Thus in the wetted-protein-colloid-particle, water forms a continuous phase and the protein substance forms the second continuous phase. The amount of water which the protein particle is able to adsorb is called the hydration capacity of the gluten. This capacity is intimately related to the internal structure of the protein particle and also to the substances existing in the solution phase in the water, as these substances affect surface tension and adsorption because of their effect on the electrical properties, hence the importance of all electrolytes, particularly those which affect the pH value.

#### Gluten Formation in Dough

There has been considerable discussion on just how gluten in dough is formed from the material in flour. The protein in wheat flour is unique in that it makes the formation of dough possible. We know that the material which constitutes the gluten is made up of several nitrogenous substances and that the gluten holds some material by simple adsorption. How is the gluten formed? The simplest way to look at it is that the protein particles make contacts and thus form a continuous phase of protein material which exists as a net or meshwork of strands. The means of union may be simply the surface forces existing in the films of water. We know that these forces are very great, especially in such thin films as exist on the surfaces of protein particles. The strength of such adherence can be seen in moistened clay. Yet as dough behaves essentially different from wetted clay or wetted starch, it appears more likely that the protein particles adhere to each other in some additional way aside from the surface tension forces in the water film. We know that one solid can adsorb another, that liquids adsorb solids and vice versa, and that the adherence may be very strong. When a dough is washed and worked in an excess of water, most of the starch is mechanically removed and there is obtained a yellowish rubbery substance known as gluten.

When flour is suspended in a relatively large amount of water this gluten formation is not observed. The protein particles of which gluten is made are in the nature of gels and of too fine a structure to be resolved even by the ultra microscope. Hence they are not observed in a suspension. These gels or protein particles exist in the flour-and-water suspension, but it is only as we get a mass of these gluten particles matted together that we can observe them. The gluten will be obtained from dough whether this is made by mixing just sufficient water to form a dough of standard stiffness and then washed and worked, or by first making a suspension and then centrifuging. The process of centrifuging is analogous to throwing down a precipitate. The process of centrifuging brings about the concentration or collection of the protein particles.

As soon as water wets the flour we may assume that the protein particles make contacts, provided the amount of water is not such as to more than fill the voids. The mixing process assists in spreading the water in a thin film over the colloid particles of the flour. The surface forces are so great that without the assistance of this mixing the water will not be spread evenly throughout the whole mass. The protein particles do not unite, forming strands, unless water is present. We know that these particles adsorb water strongly. We also know that the starch particles adsorb water, but starch alone mixed with water does not form dough. A stiff mixture of starch and water, or of clay and water may have a considerable coherence depending on the amount of water, in other words the thickness of the film, but dough is different. It behaves as though the whole mass were permeated by an elastic or rubbery network. This network is composed of the strands or chains formed by protein particles which adhere to each other.

It is evident that the strands or meshes of protein gels forming gluten must be more attenuated when they enclose the starch grains than when we have them in the gluten ball. As soon as the starch grains are removed by washing, more and firmer contacts are possible because of the greater surface areas of the protein particles exposed to each other. As soon as the starch grains are removed the protein particles become matted and form the elastic substance known as wet gluten.

Chemical analysis has shown that this gluten consists mostly of the two proteins, gliadin and glutenin. The other proteins from the flour are lost in the washing process. Gluten holds by adsorption several other substances such as ash material and cellulose. As the ratio of dry substance to water is approximately 1 to 3 it indicates some sort of



definite combination between the water and the protein material. Whether the two proteins gliadin and glutenin are combined before water is added we do not know. However, as the preharvest factors are of first importance in determining the quality of wheat, the material of the complex, gliadin and glutenin, which forms the protein particles of the dough, is formed when the endosperm is laid down in the wheat kernel.

### A Picture of the Colloid Structure of Dough

We may now picture the colloid of the dough. We have at least two continuous phases, water and the gluten meshwork. In this double continuous phase system are held the starch grains as well as the non-gluten proteins, all covered by a film of water. In this water are found molecularly dispersed salt, sugar, and simpler organic compounds such as amino acids or soluble proteins. Fat added as shortening is adsorbed at the water-protein as well as at the water-starch interface. This adsorbed layer of fat at the interfaces weakens the adherence between the protein particles and makes for lightness in bread.

Into this most complex environment we place the yeast plant. When we make a baking test we thus deal with physical, chemical, and biological forces, and the baking test becomes one of the most delicate tests we can imagine.

### Viscosity of Dough

Viscosity is the resistance offered to moving one layer of molecular units over another layer. This resistance in liquids is due to surface tension forces. In dough we may say that viscosity is due to the surface tension forces in the films of water adsorbed on the starch and protein particles. As such films of water have a thinness in the range of molecular attraction, the surface tension forces would resist a shearing movement, or the movement of one layer of molecules over another. The first layers of water molecules adsorbed on the surface of the particle would be held so firmly as to have very little or no freedom of motion. The layers outside this would possess more freedom to move over each other, as do the layers of water molecules forming the telescoping water tubes in a capillary. As successive layers of water molecules have increasing freedom of motion as they are further away from the surface of the particle, viscosity is decreased by increase in amount of liquid.

Viscosity from this standpoint exists in any mixture of water and fine particles. Minimum viscosity occurs when the mass of water is

so great that the sphere of molecular attraction surrounding each particle does not reach the sphere of its neighbor. Maximum viscosity occurs when the amount of water is such that movement can not take place without a tear or permanent deformation. Viscosity from this standpoint thus depends directly on the thickness of the film of water surrounding the particle. It is evident also that the thickness of the film of water depends on the amount of water in relation to total surface,

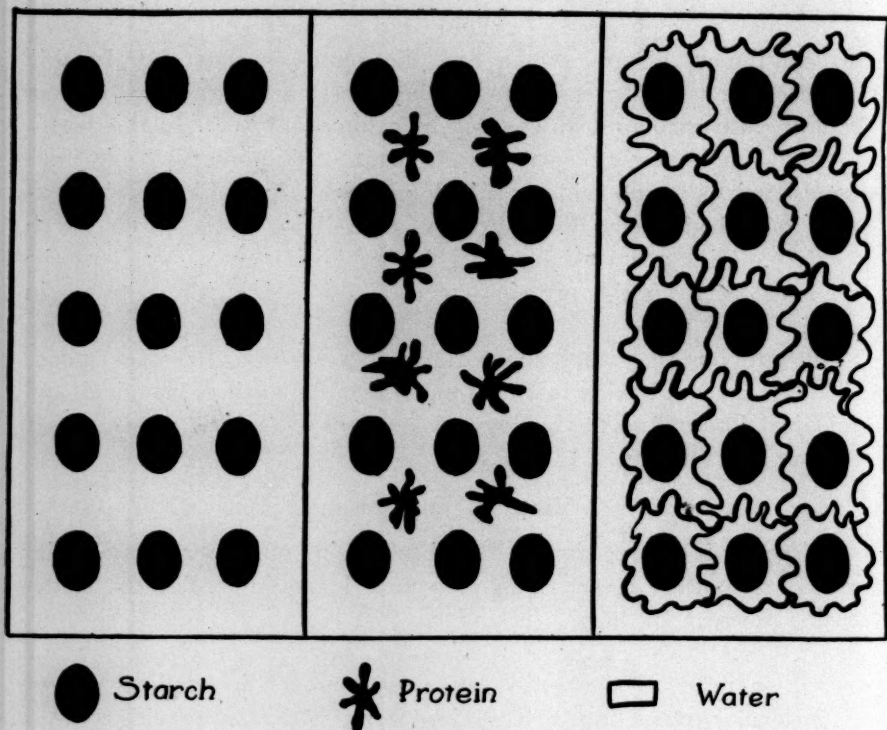


FIGURE 2

This figure is intended to illustrate the colloidal structure of dough. Starch and water form a two-phase system, the same is true of protein and water. Before the dough is formed both protein and starch are in a discontinuous phase. As soon as the dough is formed, there are two continuous phases, water and the gluten meshwork, while starch remains as a discontinuous phase. Because water wets the starch grains and because of the strength of the water films around these starch grains, starch has an influence on the colloidal quality of the dough. But dough from flour has a quality different from a mixture of starch and water. This is due to the adherence of the protein particles to each other forming strands which help to bind this whole mass together. The inherent structure of the particles forming these strands and the environment which affect adherence of the particles to each other determines the quality of dough of one flour as compared with another.

The above considerations deal with viscosity as it behaves when the particles exist in the discontinuous phase. Dough behaves as if the protein particles united to form strands, meshes, or chains. When-

ever such phenomena occur, the viscosity is enormously increased. These strands or meshes offer resistance to being torn apart and hence can act as binding material. These strands also have a large surface in proportion to the mass and hence offer opportunity for maximum action of surface tension forces. Hence the tensile strength of these strands together with the surface tension forces inherent in the adsorbed water films determine the viscosity of dough. The surface tension forces of water would not differ as much in different flours as the tensile strength of the strands or meshes formed from the protein particles. This simply means that quality in different flours is due more to differences in the structure of the protein particles and the way they are related to each than to any other factors.

### The Quality of Dough

The quality of dough is then finally determined first by the structure of strands which the protein particles form in the same way as the quality of a rope depends on the quality of the individual fibers. As starch plays a part in viscosity and as not all starches are the same, starch also plays at least some rôle in dough quality. The inherent structure of the protein particles is determined by the conditions existing when the kernel was formed and this is then the primary factor which determines quality. The factor is beyond the control of the baker. The second factor which determines the quality is the environment of the protein particle and to the extent that the baker is able to determine this environment, he is able to influence the quality of the resulting bread. The presence or absence of any substance or condition which would cause the protein particles to adhere more or less firmly in forming the strands, will affect the quality of the dough. We know that optimum precipitation of colloid particles takes place at a hydrogen-ion concentration definitely related to the isoelectric point. This means that the pH of the environment of the colloid protein particle will influence the quality of the dough. Hydrogen-ion concentration as well as other electrolytes influence hydration and this in turn affects viscosity, which is one of the important factors in dough quality. Enzyme activity, particularly proteolytic, will modify the structure of the protein particles and hence the quality of dough.

Good quality of dough involves both gas production and gas retention. Gas will be produced if the environment is suitable for rapid yeast activity. This will be obtained if the temperature is right and yeast food is present, notably sugar, either added or produced by enzyme activity. The gas is retained because of the colloid structure of the dough. The yeast plant, together with starch, is enmeshed in



the strands formed from the protein particles. The whole is covered by a film of water in which available yeast food is dissolved. As soon as evolution of gas begins, a cell is formed. This is made possible because of the surface tension forces inherent in the film of water which covers the surface of the protein strands. These have a rubbery elasticity and the cell inclosing the gas will increase in size in proportion to the production of gas. When the elastic limit is reached the cells will coalesce. If this takes place before the desired volume has been attained, the bread will be of coarse texture, which indicates weakness of gluten.

Dough quality, then, depends on the structure of the protein particles and the manner in which these unite to form strands or chains. If these protein particles are too few in number, the gluten meshes will not be strong enough no matter what the quality. This is the situation in flour of low-protein but of good quality. If the protein particles have a weak structure the gluten meshes will be weak no matter what the quantity. A weak structure will also result if the environment is such that the protein particles can not form strong contacts. The desired quality is obtained when the internal structure of the protein particles and the environment are such that strong strands are formed and when there are enough protein particles present to form enough such strands.

### Summary

The particle is the center around which ideas in colloid chemistry are organized, as the molecule is the organizing center in general chemistry. The dough is a mass of starch and protein particles, covered by thin films of water. The surface tension forces inherent in these films of water bind the starch particles together as the particles of clay are held together. These forces also help to hold the protein particles together. However, the colloidal behavior of dough can not be accounted for on this basis alone. The assumption is made that the protein particles form chains or strands which have a rubbery elasticity and these strands are matted together in a mass ordinarily known as gluten.

The quality of dough is determined by the number of particles present which form the strands, by their inherent structure and also by the environment of the particles. The number of particles is related to the quantity of protein in the flour, and their structure to the quality. These two are determined when the wheat kernel is formed. The manner of coherence among the particles and hence the quality



of the gluten strands depend also on the environment of the particles. This environment consists mostly of water and substances dissolved in this water such as sugar, enzymes, and electrolytes. Some of these substances affect the electrical condition and hence the binding force among the particles. The environment controls the conditions for yeast growth, and as a whole it is largely under the control of the baker. By modifying the environment, qualities inherent in the colloid structure may be modified to a certain limited extent. In other words, factors which determine quality are to the largest extent pre-existent in the flour before the baker gets it, but he can to a certain extent modify the quality because of his control over the environment of the protein particles.

#### HYGROSCOPIC MOISTURE OF CEREAL GRAINS AND FLAXSEED EXPOSED TO ATMOSPHERES OF DIFFERENT RELATIVE HUMIDITY

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It has long been known that cereal grains are hygroscopic and that their moisture content will vary with the conditions under which they are exposed. The moisture content of wheat stored in a dry region is much lower than in sections where the humidity of the atmosphere is greater. In the same locality the moisture content varies with the time of the year, owing to the same cause.

Upon storing 900 bushels of corn containing 14.57% of moisture at a temperature of 57° to 67° F., Duvel (1909) found a shrinkage in weight of 1.59% in the surface layer. Duvel and Duval (1911) found that corn stored in an elevator for 147 days lost 5.6% of its weight by natural shrinkage. The moisture changed from 18.8% to 14.7%. While in sound condition, the rate of shrinkage was largely influenced by its moisture content, the relative humidity of the air, and other weather conditions. These investigators, in 1913, found a natural shrinkage in commercial corn during transit, the loss varying with the water content of the grain and the atmospheric conditions to which it was exposed. They found shrinkage in corn that was hot and sour to be very rapid, amounting to several per cent within a few days.

According to McHargue (1920), this is caused by the rapid deterioration of oil, sugars, and starches in the germ of the corn.

The Utah Agricultural Experiment Station (1914) stored wheat and oats, grown under dry farming and irrigated conditions, for two years. The wheat gained from 0.61 to 2.75% in moisture during this time. The oats gained 2.12 to 2.32% of moisture. During the spring and summer months and into the fall, there was a loss of moisture and weight, but there was always more moisture present than at threshing time. The grain increased in weight again during the winter. There seemed to be little difference in the change in moisture content in the grain raised under irrigation and that raised under dry-farming conditions. The method of harvesting had no influence on the change in weight. It was thought that the stage of maturity and dryness at harvest are the chief factors that determine changes in weight during the first fall and winter after harvesting. The changes in weight are attributed to moisture gains and losses.

The Ohio Agricultural Experiment Station (1918) also stored oats for five years. During this period, the grain gained and lost moisture, depending upon the season of the year. The Michigan station records losses (1901) in weight of 0.2 to 3.4% in from five to seven months.

The Illinois Agricultural Experiment Station (1915) reports figures showing that wheat stored from 10 days to 3 years suffered an average loss of 2.32%. It was estimated that in large elevators the shrinkage in wheat would amount to from 1 to 2% in six months.

The Ohio station (1918) records a net loss of 0.41% in the weight of 40 bushels of wheat stored for five years. During one season, the grain increased in weight 1.08%.

During the crop years 1914 to 1919, Baldwin (1921) made studies of the moisture content of the various commercial classes of wheat received at Portland, Ore., from interior points. Comparisons of these tests were made with temperature and relative humidity figures. From the time the new crop began to reach Portland, in August, until the following March and April, there was a gradual rise in the moisture content of the wheat, when with a lowering in the relative humidity and a rise in temperature, the wheat began to lose some of the moisture it had absorbed during the winter months.

Investigations were likewise made regarding the change in moisture of lots of wheat stored in sacks on docks in Portland during the crop years 1917 and 1918. The average of 87 lots of wheat showed a gain of 1.6% in moisture. This is about the same gain obtained at interior points during the winter months.

Stockham (1917) says that the moisture content for different wheats and their various mill products is variable even under the same atmospheric conditions.

Boerner (1915) reports data which show that the shrinkage of ear corn on the farm varies considerably in different kinds of storage cribs and different seasons of the year, and with different kinds of corn, so that no very accurate data can be given that will apply to all corn.

Results from 2267 tests showed that corn taken from cribs in south central Illinois averaged 21.7% of moisture in November and 12.2% in June, which is equal to a loss of moisture of 9.5%, and shrinkage in weight of 10.8%. Shrinkage in the cob progressed much faster than did shrinkage in the kernel.

McHargue (1920) states that corn containing 12% moisture will readily absorb more when exposed to conditions in which free moisture is present.

Bailey (1921) records a series of experiments in which he studied the moisture content of shelled corn when in equilibrium with atmospheres of different relative humidity. The hygroscopic moisture varied from 8.25% at 34.8% relative humidity (75° C.) to 17.57% at 85.4% relative humidity, showing conclusively that the moisture in corn may undergo considerable change when exposed to atmospheres of different humidity.

In none of these experiments save those of Bailey (1921) and Stockham (1917) have humidity conditions been controlled throughout the period of exposure.

It seemed desirable, therefore, to ascertain the moisture content of cereal grains and flaxseed in atmospheres of differing but constant humidity, and after a period of exposure sufficient to permit the hygroscopic moisture of the grains studied to reach equilibrium with the atmosphere.

Such data would be of service in several ways. Shippers, buyers, and grain inspection officials need more precise information concerning the changes in moisture and consequent net weights of the various cereal grains. Elevators and conditioning houses, if the percentage of moisture in any given cereal is known at a definite percentage of humidity and degree of temperature, can use the data in conjunction with the specific heat of the grain, for working out plans to dry or moisten grains as they lie in the bin. Shrinkage or increase of weight in grain is also of interest to the grain dealer as it is a matter of concern to him to know whether the conditions under which his grain is stored or transported will result in increase or decrease of its weight.

### Method of Procedure

The humidity of the atmosphere to which the grain was exposed was conditioned by the method of Wilson (1921). This method was later checked by the exposure to the vapor pressure of sulphuric acid solutions in desiccators.

Seven solutions were prepared to give humidities of 15°, 30°, 45°, 60°, 75°, 90°, and 100° respectively. The temperature was about 25° C., varying a few degrees above this. This is not of importance, however. As has been pointed out by Wilson (1921), it is ordinarily not necessary to control the temperature accurately, as variations of 5° or 10° C. have practically no effect on humidity equilibria.

For determining the humidity equilibria of the grain under test, the apparatus as described by Wilson (1921) was also used. The capacity of each wash bottle was 1000 cubic centimeters. The U-tube employed was 8 inches high and 1 inch in diameter. In making the tests, approximately 60 grams of seed was placed in a previously weighed tube, the whole reweighed, and placed in position in the train. Compressed air of the relative humidity values given above was then passed through the apparatus at the rate of about 200 cc. per minute. Preliminary studies indicated that it required from six to eight days for the grain to reach equilibrium in moisture content. To insure that ample time was given to reach equilibrium, the tubes were weighed every 24 hours, and continued in position until two 24-hour weighings were identical. The contents of the tubes were then quickly removed to friction-top moisture dishes, and the moisture was determined by heating in a water oven at a temperature of 100° C. to a constant loss in weight (120 hours). The moisture content of the grain before use in the tests was also determined by the water-oven method in order to determine the original dry weight. At the end of each test portions of the sulphuric acid in the wash bottles were tested in order to determine whether any change had taken place during the test run. Small variations were found but seldom of a magnitude greater than one-half of 1 per cent. Results are expressed as per cent of the dry weight.

Tables I to IV and summary Table V record the percentages of moisture in the commercial classes of corn, oats, barley, buckwheat, flaxseed, rice, rye, and wheat, when exposed to atmospheres of approximately 15, 30, 45, 60, 75, 90, and 100% relative humidity at 25° to 28° C. The same data are recorded graphically in Figure 1.



TABLE I  
HYGROSCOPIC MOISTURE OF WHEAT EXPOSED TO ATMOSPHERES OF DIFFERENT RELATIVE HUMIDITY AT 25° TO 28° C.

Relative humidity of atmosphere at 25° to 28° C.		Hygroscopic Moisture Sample numbers										Average	
Per ct.	Per ct.	1	2	3	4	5	6	7	8	9	10	11	Per ct.
15	7.10	6.83	6.91	6.85	6.85	6.69	6.92	6.98	6.65	6.68	6.99	7.18	6.89
30	9.40	9.46	9.20	9.19	9.60	8.94	8.94	8.90	9.32	9.50	9.28	9.59	9.30
45	11.86	11.48	11.87	11.46	12.18	11.56	11.56	11.45	12.13	11.86	11.96	11.60	11.76
60	14.51	14.02	14.28	14.40	14.16	14.44	14.42	14.42	14.49	14.01	14.01	13.69	14.27
75	17.95	17.69	17.30	16.80	17.33	17.27	17.05	17.05	16.72	17.22	16.86	17.33	17.13
90	25.24	25.59	24.96	25.77	24.61	25.26	25.33	25.49	24.85	25.26	24.70	25.16	25.16
100	34.00	35.03	34.12	33.43	32.64	34.03	34.28	35.61	32.49	33.71	33.71	33.77	33.95
% Protein, dry basis		9.46	9.76	10.23	11.15	12.62	12.80	13.23	14.08	15.12	15.99	11.75	.....
% D. H. & V. kernels*		45.3	50.0	52.6	89.4	91.0	91.2	89.8	91.0	94.2	91.4	95.0	.....
Hard Red Spring Wheats													
15	7.94	7.21	6.93	7.03	.....	.....	.....	.....	.....	.....	.....	.....	7.27
30	9.79	9.14	8.64	9.43	.....	.....	.....	.....	.....	.....	.....	.....	9.25
45	11.44	11.21	11.08	11.12	.....	.....	.....	.....	.....	.....	.....	.....	11.21
60	13.96	13.46	13.32	12.86	.....	.....	.....	.....	.....	.....	.....	.....	13.40
75	17.47	17.54	17.54	16.74	.....	.....	.....	.....	.....	.....	.....	.....	17.32
90	24.92	23.65	24.65	25.23	.....	.....	.....	.....	.....	.....	.....	.....	24.61
100	.....	33.65	34.27	32.35	.....	.....	.....	.....	.....	.....	.....	.....	33.42
White Wheats													
15	7.40	6.68	7.78	.....	.....	.....	.....	.....	.....	.....	.....	.....	7.25
30	9.74	9.59	9.91	.....	.....	.....	.....	.....	.....	.....	.....	.....	9.41
45	10.77	11.17	10.93	.....	.....	.....	.....	.....	.....	.....	.....	.....	10.96
60	13.15	13.53	13.33	.....	.....	.....	.....	.....	.....	.....	.....	.....	13.34
75	17.49	17.44	17.87	.....	.....	.....	.....	.....	.....	.....	.....	.....	17.60
90	24.69	25.22	23.69	.....	.....	.....	.....	.....	.....	.....	.....	.....	24.53
100	34.89	35.60	36.65	.....	.....	.....	.....	.....	.....	.....	.....	.....	35.71

\*Dark, hard and vitreous kernels.

TABLE I—Continued

Relative humidity of atmosphere at 25° to 28° C.		Hygroscopic Moisture Sample numbers											
1	2	3	4	Durum Wheats				7	8	9	10	11	Average
Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.
15	6.86	7.26	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	7.06
30	9.34	9.18	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	9.26
45	11.53	10.82	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	11.18
60	13.38	12.59	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	12.98
75	16.94	15.97	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	16.45
90	24.37	23.54	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	23.96
100	35.39	37.29	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	36.34
Soft Red Winter Wheats													
15	6.74	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	6.74
30	9.46	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	9.46
45	11.82	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	11.82
60	13.49	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	13.49
75	17.09	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	17.09
90	24.59	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	24.59
100	34.47	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	34.47

### Hygroscopic Moisture of Wheat

Hygroscopic studies were made on eleven samples of Hard Red Winter wheat, four samples of Hard Red Spring, three of the Common White, two of Durum, and one of the Red Winter class of wheats. All the wheat samples weighed approximately 60.5 pounds to the Winchester bushel. The Hard Red Winter wheats were chosen with protein content varying from 9.46% to 15.66%, dry-matter basis. The texture of the wheat varied as shown in Table I.

From the data given in Table I it is shown that wheat in equilibrium with different atmospheric humidity at 25° to 28° C. ranges from a little more than 6.5% of moisture at 15% relative humidity to as much as 36% at 100% relative humidity. This last value is not far different from those reported by Stockham (1917) for wheat exposed to a saturated atmosphere. There are differences in the hygroscopic moisture of individual samples of wheat when exposed to atmospheres of different relative humidity, but on the average the hygroscopic moisture of all the classes of wheat studied was closely alike.

An increase in the protein content of the wheat from 9.46 to 15.66% seemed to exert no influence on the hygroscopic condition of the wheat. This is likewise true of samples having high and low percentages of dark, hard, and vitreous kernels. Within the spring wheat group, samples 2 and 3 contained 100% of dark, hard, and vitreous kernels, and 100% of starchy kernels, respectively, both types of kernels were selected from the same bulk sample. The difference in hygroscopic moisture within each of these types, at all degrees of relative humidity below 90, was practically negligible.

It is clear also that hygroscopic moisture does not increase at a uniform rate when in equilibrium with an increasing atmospheric humidity, a much greater change in hygroscopic moisture being recorded with a change in relative humidity from 75 to 90 or from 90 to 100% relative humidity than from 45 to 60 or 60 to 75% relative humidity.

### Hygroscopic Moisture of Corn and Rye

In Table II are listed data obtained from a study of the hygroscopic moisture of corn and rye, when exposed to different degrees of relative humidity. Two samples of dent corn and one of popcorn, and three of rye were used in this series of tests

TABLE II

HYGROSCOPIC MOISTURE OF CORN AND RYE EXPOSED TO ATMOSPHERES OF DIFFERENT RELATIVE HUMIDITY AT 25° TO 28° C.

Relative humidity of atmosphere at 25° to 28° C.	Hygroscopic moisture			
	Sample number			
	1	2	3	
	White Dent	Corn Yellow Dent	Pop-corn	Average
Per ct.	Per ct.	Per ct.	Per ct.	Per ct.
15	7.06	6.88	7.35	7.09
30	9.24	9.17	9.34	9.25
45	11.58	11.70	10.88	11.39
60	14.83	14.85	13.89	14.52
75	17.22	17.35	15.80	16.79
90	23.35	23.55	22.47	23.12
100	32.64	31.20	29.83	31.22
		Rye		
15	7.07	7.63	7.79	7.49
30	9.21	9.69	9.83	9.58
45	10.94	11.99	12.22	11.72
60	13.57	14.12	14.12	13.94
75	16.84	17.73	17.66	17.41
90	25.40	26.21	26.14	25.92
100	37.03	35.80	36.70	36.51

Considering the samples of corn, practically no difference was found with regard to the hygroscopic moisture of the two samples of dent corn studied, at all percentages of relative humidity, 100% relative humidity excepted. With the popcorn, a decidedly lower percentage of hygroscopic moisture was found at all percentages of relative humidity. Whether the variety difference in this connection is of significance is not determinable from the data at hand. Bailey (l. c.) has pointed out departures from the condition obtained with dent corn with a sample of sweet corn.

The average hygroscopic moisture of rye is somewhat similar to that of wheat, at the lower percentages of relative humidity studied, becoming somewhat higher at 45° of relative humidity and above.

#### Hygroscopic Moisture of Barley, Buckwheat, Oats, and Rice

The data on the hygroscopic moisture of the coarse grain exposed to different relative humidity as described above, is given in Table III. Tests on barley and buckwheat result in figures identical with those obtained with some of the wheat samples previously tested.

Oats and rice, on the basis of averages as well as on the basis of individual samples, contain less hygroscopic moisture than any of the other cereals studied, under all conditions of atmospheric humidity. These two cereals contain on the average 1 to 1.5% less moisture under all conditions of relative humidity.



TABLE III

HYGROSCOPIC MOISTURE OF BARLEY, OATS, RICE, AND BUCKWHEAT EXPOSED TO ATMOSPHERES OF DIFFERENT RELATIVE HUMIDITY AT 25° TO 28° C.

Relative humidity of atmosphere at 25° to 28° C.	Hygroscopic moisture			
	1	Sample number 2 Barley	3	Average
Per ct.	Per ct.	Per ct.	Per ct.	Per ct.
15	6.67	6.39	6.29	6.45
30	9.22	9.26	9.24	9.24
45	11.55	10.98	10.96	11.16
60	13.95	13.63	13.63	13.74
75	17.16	16.55	16.94	16.88
90	24.96	23.16	24.48	24.17
100	37.01	34.32	38.62	36.65
		Oats		
15	6.30	5.79	5.99	6.03
30	9.00	8.67	8.60	8.76
45	10.66	10.52	10.63	10.60
60	13.30	13.34	13.58	13.40
75	16.56	15.67	15.93	16.05
90	23.52	22.56	21.86	22.65
100	32.80	31.65	30.70	31.71
		Rice		
15	7.05	7.21	7.57	7.27
30	9.90	9.66	10.19	9.92
45	11.97	12.16	11.87	12.00
60	15.06	14.67	13.48	14.38
75	17.13	16.69	16.75	16.86
90	22.73	21.94	21.77	22.15
100	31.93	30.03	30.59	30.85
		Buckwheat		
15	7.17	.....	.....	7.17
30	9.99	.....	.....	9.99
45	12.18	.....	.....	12.18
60	14.52	.....	.....	14.52
75	17.61	.....	.....	17.61
90	23.63	.....	.....	23.63
100	32.47	.....	.....	32.47

### Hygroscopic Moisture of Flaxseed

Flaxseed, of course, is not a cereal. It is distinctively different from the cereals in that it is principally oil bearing in nature. Oil, moreover, has little or no capacity for water. It is to be expected, therefore, that as only about 60% of the flaxseed kernel can be occupied by moisture, the hygroscopic moisture of this type of seed should be different from that in the cereal grains.

Six samples of flaxseed, representing the four commercial classes, were studied, the data obtained being recorded in Table IV.

Compared with the cereal grains, the hygroscopic moisture of flaxseed when exposed to different atmospheres of relative humidity is

uniformly lower. It will vary from a little more than 4% at a relative humidity of 15% to a little less than 30% at a relative humidity of 100%. As with the cereal grains, the increase in moisture content with increasing atmospheres of relative humidity is not uniform, increasing with greater steps at the higher percentages of relative humidity.

It is evident that the percentage of moisture in cereals and flaxseed may undergo considerable change when exposed to atmospheres of different relative humidity, providing of course that the conditions of exposure are such that movement of water vapor can take place. This in turn depends upon the temperature of the grain and the outside air, the extent and efficiency of air circulation, the size, shape, and composition of the containers in which the grain is housed, and other factors.

TABLE IV  
HYGROSCOPIC MOISTURE OF FLAXSEED EXPOSED TO ATMOSPHERES OF DIFFERENT RELATIVE HUMIDITY AT 25° TO 28° C.

Relative humidity of atmosphere at 25° to 28° C.	Hygroscopic moisture						Average
	North-western grown seed	South-western grown seed	Argentine seed	Indian seed	Fiber flaxseed	Man-churian	
Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.
15	4.46	4.68	4.80	4.05	5.15	4.82	4.66
30	5.56	5.86	6.27	5.15	6.38	6.29	5.91
45	6.45	6.39	7.29	5.95	7.07	7.33	6.74
60	8.62	8.43	9.15	7.50	8.82	9.20	8.62
75	11.43	11.51	11.42	9.60	11.48	11.47	11.15
90	17.91	18.44	18.11	16.20	18.22	18.56	17.90
100	25.39	28.65	27.66	25.54	29.37	27.71	27.29
% oil, dry basis	40.30	38.43	42.32	42.99	39.41	42.14	.....

Surface shrinkage in stored grain is common. Duvel (l. c.) reports marked changes in the moisture content of the surface layers of corn stored in elevators or in freight cars on track or in transit, the extent of drying depending upon the condition of the grain and the temperature and humidity of the surrounding atmosphere. Changes in the moisture content of grain along the walls of wooden and concrete storage tanks has frequently been observed. In wooden tanks, these moisture changes are largely due to absorption phenomena, while in concrete tanks changes in moisture are due to capillary losses of moisture through the concrete walls. That movement of water actually takes place through concrete walls is shown in Technical Paper No. 3, United States Bureau of Standards.

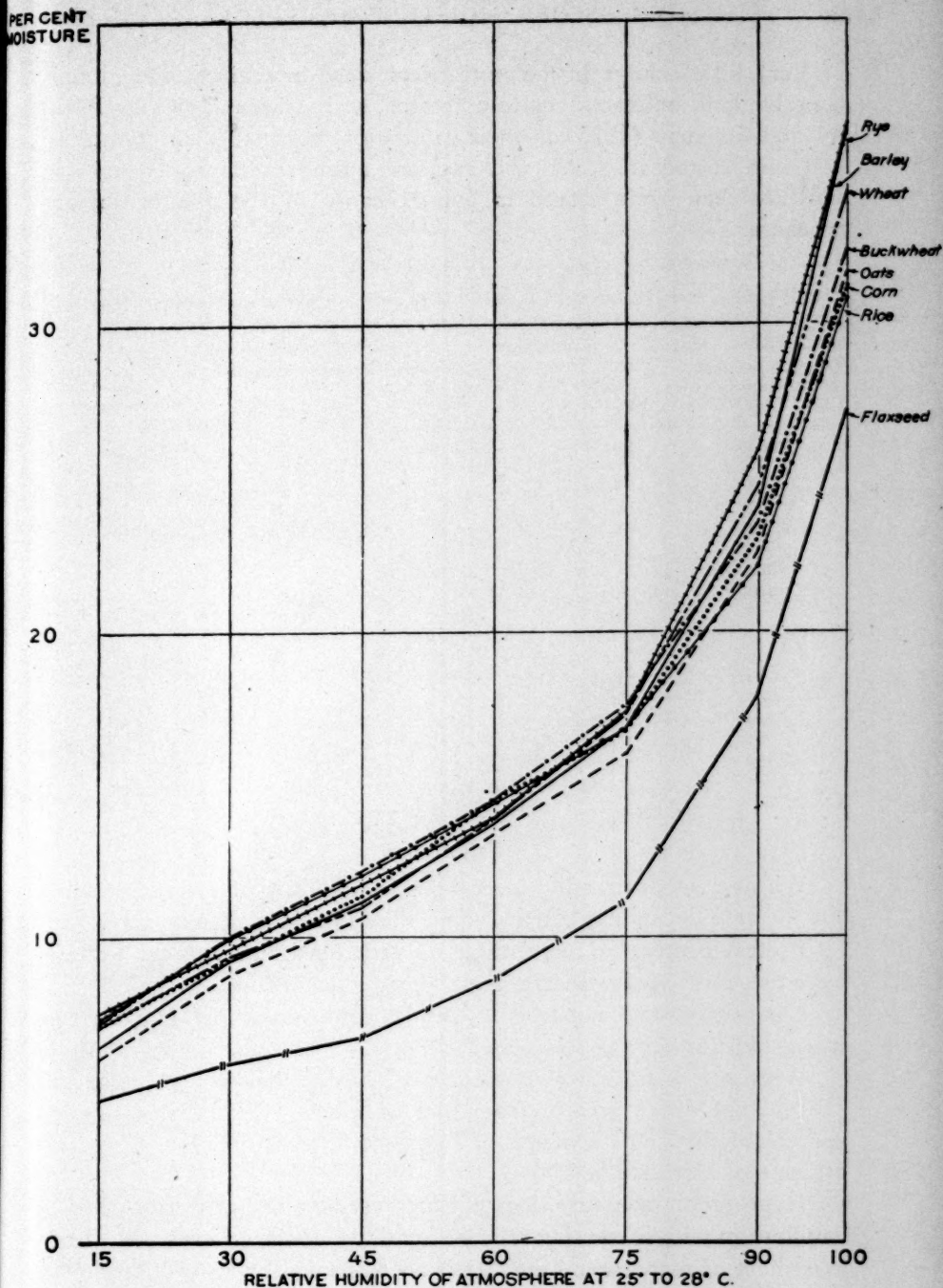


Fig. 1. Hygroscopic Moisture of Cereal Grains and Flaxseed Exposed to Atmospheres of Different Relative Humidity at 25° to 28° C.

Very little change in moisture takes place in grain stored in considerable bulk unless extensively areated, as has been shown by Rothgeb and Coleman (1923) in their ventilation work on farm grain bins.

Grain stored in sacks will respond more readily to changes in moisture than grain stored in bulk, because of the greater surface exposure.

TABLE V  
HYGROSCOPIC MOISTURE OF CEREAL GRAINS AND FLAXSEED EXPOSED TO ATMOSPHERES OF DIFFERENT RELATIVE HUMIDITY AT 25° TO 28° C.

Relative humidity of atmosphere at 25° to 28° C.	Barley 3 samples	Average hygroscopic moisture Buckwheat 1 sample	Corn 3 samples	Flaxseed 6 samples
Per ct.	Per ct.	Per ct.	Per ct.	Per ct.
15	6.45	7.17	7.09	4.66
30	9.24	9.99	9.25	5.91
45	11.16	12.18	11.39	6.74
60	13.74	14.52	14.52	8.62
75	16.88	17.61	16.79	11.15
90	24.17	23.63	23.12	17.90
100	36.65	32.47	31.22	27.29
	Oats	Rice	Rye	Wheat
	3 samples	3 samples	3 samples	11 samples
15	6.03	7.27	7.49	7.03
30	8.76	9.92	9.58	9.34
45	10.60	12.00	11.72	10.99
60	13.40	14.38	13.94	13.81
75	16.05	16.86	17.41	17.30
90	22.65	22.15	25.92	24.82
100	31.71	30.85	36.51	34.38

### Summary

Wheat responds readily to changes in humidity of the surrounding air, the rate depending entirely upon the conditions of exposure.

Protein content, or percentage of dark hard and vitreous kernels, were not correlated with the hygroscopic moisture.

The hygroscopic moisture of samples of different market classes of wheat did not vary appreciably.

Hygroscopic moisture in corn, oats, barley, buckwheat, rice, and rye did not differ greatly from that of wheat. Flaxseed contained appreciably lower percentages of hygroscopic moisture than the cereal grains that were studied.

Hygroscopic moisture does not increase at a uniform rate when in equilibrium with increasing relative humidity of the atmosphere. Each increment of increase in humidity is accompanied by an acceleration in the rate of increase in content of hygroscopic moisture. The curve resulting from plotting humidity as abscissas against hygroscopic moisture as ordinates takes the form of a parabola.



## Literature Cited

- Bailey, C. H.  
1921. Respiration of shelled corn. Minn. Agr. Exp. Sta. Tech. Bul. 3.
- Baldwin, R. L.  
1921. U. S. D. A. Federal grain supervision project letter, Vol. 2, No. 25.  
1921. U. S. D. A. Federal grain supervision project letter, Vol. 2, No. 33.
- Boerner, E. G.  
1915. Shrinkage and price of ear corn. U. S. D. A. Grain Standardization Recorder, Vol. 3, p. 51.
- Burlison, W. L., and Allyn, P. M.  
1915. Prices and shrinkage of farm grain. Ill. Agr. Exp. Sta. Bul. 183.
- Duvel, J. W. T.  
1909. The deterioration of corn in storage. U. S. Bureau of Plant Industry Circ. 43.
- Duvel, J. W. T., and Duval, Laurel  
1911. The shrinkage of corn in storage. U. S. Bureau of Plant Industry Circ. 81.  
1913. The shrinkage of shelled corn while in transit. U. S. D. A. Bul. 48.
- Harris, F. S. and Thomas, George  
1914. The change in weight of grain in arid regions during storage. Utah Agr. Exp. Sta. Bul. 130.
- McHargue, J. S.  
1920. The cause of deterioration and spoiling of corn and cornmeal. Jour. Ind. Eng. Chem. Vol. 12, pp. 257-262.
- Rothgeb, B. E., and Coleman, D. A.  
1923. Ventilated farm grain bins. U. S. G. S. A., G. I. 24.
- Smith, C. D.  
1901. Shrinkage of farm products. Mich. Agr. Exp. Sta. Bul. 191.
- Stockham, W. L.  
1917. The capacity of wheat and mill products for moisture. N. D. Agr. Exp. Sta. Bul. 120, p. 108.
- Welton, F. A.  
1918. Ohio Agr. Exp. Sta. Monthly Bul. Vol. 3, No. 2.
- Wig, R. J., and Bates, P. H.  
1911. Tests of the absorptive and permeable properties of Portland cement mortars and concretes, together with tests of damp proofing and waterproofing compounds and materials. Tech. Paper No. 3. U. S. Bureau of Standards.
- Wilson, R. E.  
1921. Humidity control by means of sulphuric acid solution with critical compilation of vapor pressure data. Jour. Ind. and Eng. Chem., Vol. 13, No. 4, p. 326.

## EFFECT OF CLIMATE AND OTHER FACTORS ON THE PROTEIN CONTENT OF NORTH DAKOTA WHEAT

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(Read at the convention June 4, 1925)

North Dakota is noted for the production of high protein wheat. While the wheat crops of the state usually average quite high in protein content, the seasonal variation is considerable. Considering the present extensive use of the protein test in buying and selling wheat on the Minneapolis market, a study of the factors affecting the protein content of wheat is of interest to both the wheat grower and the miller.

### Crop Surveys and Protein Variation

The wheat crop of 1921 was quite high in protein content, and the average of all samples analyzed was 15.02%. The crop of 1922, however, was below the average in protein content, and owing to the shortage of high-protein wheat, premiums were offered on the Minneapolis market for high-protein content. The North Dakota Experiment Station in co-operation with the county agents of the state, made a survey of the 1922 crop for protein content, and the average of all samples collected in 1922 was 12.00%. This survey was repeated in 1923 and 1924. The 1923 crop averaged 13.29% protein, but the crop of 1924 averaged only 11.33%, the lowest for the four years. The wheat crops for these four years, therefore, showed considerable variation in protein content.

Other information obtained through these surveys may be summed up as follows: (1) No area within the state, except a small area around Rugby, Pierce County, produced wheat of high-protein content consistently for the three years. (2) Samples collected from limited areas, as a single county, showed considerable variation in protein content. (3) Some high-protein wheat was found each season, and high-protein samples were often found in areas where the general average was low. (4) Wheat following such legume crops as sweet clover, was usually higher in protein content than other samples from the same area.

### Effect of Climate on Protein Content of Wheat

It is generally recognized that climate is a very important factor in determining the protein content of wheat. The principal hard-wheat producing regions of the North American continent are the northwest and the southwest great plains states of the United States and the

Canadian provinces of Manitoba, Saskatchewan, and Alberta. All these regions have a comparatively low annual rainfall.

The celebrated "tri-local" experiments conducted by Le Clerc (1910), of the United States Department of Agriculture, co-operating with the Kansas and California stations showed that climatic conditions were more important than seed or soil in determining the protein content of wheat. He states that "wheat of any one variety, from any one source, and absolutely alike in chemical and physical characteristics, when grown in different localities, possessing different climatic conditions, yields crops of very widely different appearance, and very different in chemical composition."

Thatcher (1913) found that climate was the chief factor in fixing the composition of the wheat crop in any given season or locality. He presents evidence that high temperatures during ripening produce high-protein wheat, and concludes also that the length of the period of kernel formation rather than that of the whole growing period determines the composition of the grain. He finds that the average weight of the kernel varies directly with the length of the development period, and that the percentage of nitrogen in the grain varies inversely with the length of this period.

Thatcher also found that the protein content of wheat from different parts of Washington varies inversely with the annual precipitation of the region.

Shutt (1910) found that a high water content of soil decreases protein content, and prolonged vegetative growth increases starch content. Early ripening, lessening of soil moisture, and high temperature produced hard high-gluten wheat. Wheat from irrigated land was lower in protein content than the same variety from non-irrigated land.

The results of most investigations indicate that excess moisture causes low-protein wheat. Stewart and Hirst (1913), of the Utah station, find that the protein content of wheat is decreased by irrigation, and that the protein content of wheat grown by dry farming averages higher than that of wheat grown on irrigated land. Greaves and Carter (1923) in a later report state that wheat decreased in nitrogen content as the irrigation water used in their growth was increased.

A review of the literature on the effect of climate indicates three factors of importance: (1) Rainfall or irrigation; (2) temperature during the growing season, and (3) length of growing season.

### Relation of Climate to Seasonal Variation in Protein Content of North Dakota Wheat

Low rainfall characterizes the climate of the principal hard wheat producing regions, and for this reason it was assumed that seasonal variation in rainfall would explain the variation in protein content. An examination of the United States weather bureau records, however, shows that we can not explain the variation in protein content during the last four years by the variation in annual rainfall.

Table I shows the mean daily temperatures for May, June, and July, rainfall and average protein content of the wheat crops for 1921 to 1924. The rainfall during these four years shows no marked variation. For June and July the highest precipitation is found in 1921 and 1923, the high-protein years, and precipitation for six months (March to August) is highest in 1921. As excess moisture tends to decrease protein content, the difference in average protein content of these four crops can not be explained by variation in rainfall.

TABLE I  
TEMPERATURE, RAINFALL,\* AND AVERAGE PROTEIN CONTENT OF WHEAT CROP, 1921 TO 1924

Year	Average protein content of crop	Mean daily temperature			Precipitation Six months March- August		
		May	June	July	June and July	March- August	Annual
		Deg.	Deg.	Deg.	In.	In.	In.
Normal.....		52.6	62.8	67.5	6.11	13.15	17.94
1921.....	15.02	52.8	68.2	71.4	6.58	13.84	19.59
1922.....	12.01	56.3	63.5	65.6	6.21	12.32	19.75
1923.....	13.35	54.1	66.1	71.2	6.96	12.49	17.76
1924.....	11.33	46.9	58.6	65.5	6.12	11.56	17.55

\*Data on rainfall and temperature from records of United States Weather Bureau.

A further examination of Table I, however, shows that the June and July mean daily temperature was above normal in 1921 and 1923. The July temperature was subnormal in 1922; and in 1924, May, June, and July were decidedly subnormal in temperature.

The relation of temperature to protein content is shown more clearly by Figure I. In 1921, abnormal June and July temperatures produced a crop of very high average protein content. The same conditions prevailed in 1923, but the June temperature was not extreme, and the average protein content of the crop, while high, was considerably lower than that of the 1921 crop.

The growing season of 1922 was characterized by a subnormal July temperature and the average protein content of the crop was much lower than in 1921 or 1923. The very low protein content of the 1924 crop was evidently due to a combination of subnormal May, June, and



July temperatures, practically the whole growing season being subnormal in temperature.

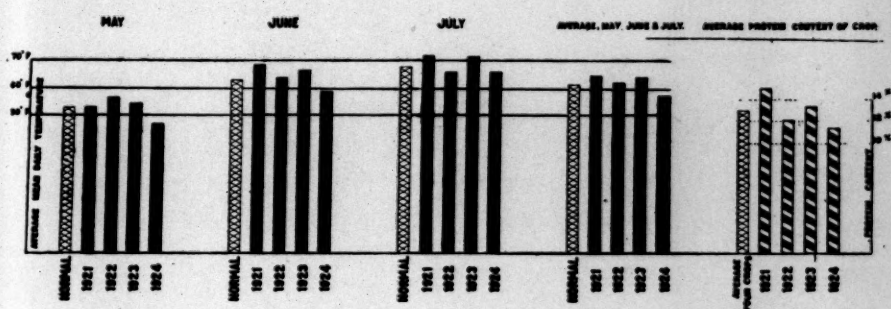


Fig. 1. Relation of mean daily temperature to protein content of bread wheats.

The difference in average protein content of the wheat crop for the four years 1921 to 1924, therefore, may be explained by the variation in mean daily temperature. As the lowest average protein content and the lowest rainfall are found in the same year (1924), the low protein content can not be ascribed to excess rainfall. The temperature of the growing season has apparently been the chief climatic factor in determining the protein content of the crops for these four years.

The 1915 wheat crop was also quite low in protein content, and the Weather Bureau records show that the mean daily temperature was subnormal in June and July, 1915. The high protein content of the 1917 crop was evidently due in part to the very low rainfall of that year.

While the subnormal temperature during the growing season in 1922 and 1924 produced low-protein wheat, it should be noted that such conditions also are favorable to high yields. Table II shows the average yield per acre and the June and July temperatures for 1910 to 1924. The years 1912, 1915, 1918, 1922, and 1924 show significantly higher yields per acre and all seasons except 1922 show subnormal temperatures for both June and July, and 1922 shows a subnormal temperature for July. Cool weather during the growing season may lower the protein content of the wheat, but the wheat grower is probably more than compensated for any discount in price due to the low protein content, by the increased yield per acre.

#### Relation of Length of Growing Season to Protein Content

Thatcher (1913), of the Washington station, found that the protein content of wheat varied inversely as the length of the growing season, and other investigators have pointed out the relation between rapid ripening and high protein content.

TABLE II

RELATION OF JUNE AND JULY TEMPERATURE TO AVERAGE YIELD PER ACRE OF WHEAT  
IN NORTH DAKOTA, 1910 TO 1924

Year	Average yield per acre* Bu.	Mean daily tempera- ture, June	Mean daily tempera- ture, July
		Deg.	Deg.
1910.....	5.0	67.3	70.0
1911.....	8.0	66.9	65.1
1912.....	18.0	61.8	66.0
1913.....	10.5	65.8	65.6
1914.....	11.2	62.2	72.1
1915.....	18.2	56.7	62.3
1916.....	5.5	58.0	73.1
1917.....	8.0	59.7	70.9
1918.....	13.6	63.3	65.5
1919.....	6.9	67.2	71.2
1920.....	9.0	62.2	68.3
1921.....	8.5	68.2	71.4
1922.....	14.1	63.5	65.6
1923.....	7.1	66.1	71.2
1924.....	15.5†	58.6	65.5
Normal.....	.....	62.8	67.5

\*Data from United States Department of Agriculture.

†Preliminary figure.

Figures 2 and 3 show that cool weather lengthens the growing season, while warm weather has the opposite effect. Figure I showed that the protein content varied directly as the June and July temperature, and Figures 2 and 3 show the same relation between June and July temperature and protein content. The protein content varies inversely as the length of the growing season.

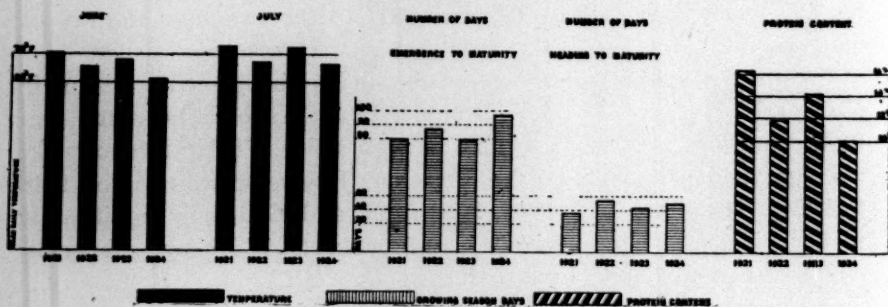


Fig. 2. Relation of mean daily temperature to length of growing period and protein content of Marquis wheat, at Fargo, N. D.

Figures 2 and 3 indicate that the total growing season from emergence to maturity is a more important factor in relation to protein content than the period of kernel development or the number of days from heading to maturity.

The effect of warm weather in hastening the maturity of crops is well known, but how can we explain the effect of temperature on

protein content? The cool weather prolongs the growing period; the upper part of the stems remain green, and the elaboration of carbohydrates by the plant continues. The protein content is limited by the available nitrates in the soil, and owing, possibly, to a lack of nitrogen, the elaboration of carbohydrates proceeds more rapidly than that of protein.

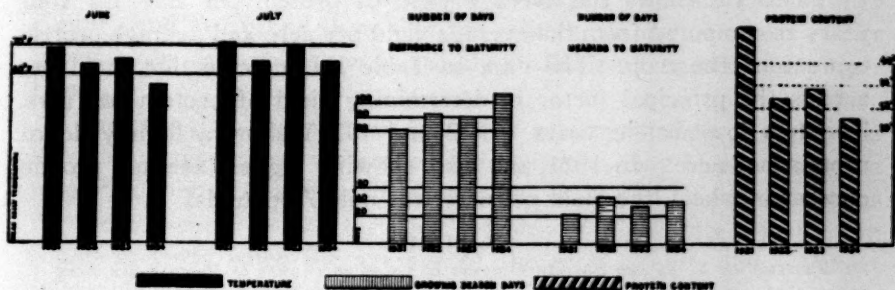


Fig. 3. Relation of mean daily temperature to length of growing season and protein content of Marquis wheat, at Dickinson, N. D.

Warm weather, however, shortens the growing period and the stems and leaves lose their green color much sooner and become browned. When the green color due to chlorophyll disappears, the plant can no longer elaborate starch.

#### Variation in Rainfall

Variation in rainfall is evidently not as great a factor in causing seasonal variation in the protein content of North Dakota wheat as the mean daily temperature in June and July. A large increase in rainfall would no doubt affect the composition of the wheat.

Comparisons made between irrigated and non-irrigated land generally show a higher protein content for wheat produced on non-irrigated land.

Considerable variation of protein content is found within limited areas. This variation is due in part to soil fertility, but may also be due to difference in rainfall during summer months. Summer showers are largely local and considerable difference may be found in precipitation in relatively small areas.

In summing up the effect of climatic variation, it appears that under North Dakota conditions variation in mean daily temperature during the growing season, particularly June and July, is largely responsible for seasonal variation in protein content of wheat. The length of the growing season as a factor is distinctly related to temperature and the effect of length of growing season is probably indirectly the effect of temperature. Considering the state as a whole, variation in rainfall is not an important factor, altho an abnormally

low rainfall may increase protein content. Variation within a crop year, however, in limited areas, may be due to variation in local rainfall during the summer months, but this variation is more probably due to variation in soil fertility.

### Yield of Protein Per Acre

Table III shows the average yield of protein per acre for four years as computed from the average yield per acre and average protein content of the crop. The data in Table III indicate that yield per acre is the principal factor in determining yield of protein per acre. The two low-protein years (1922 and 1924) show a high yield of protein per acre. In 1921, the year showing highest average protein content in wheat, the yield per acre was only 77 pounds.

TABLE III  
RELATION OF YIELD AND PROTEIN CONTENT OF CROPS TO POUNDS OF PROTEIN PER ACRE

Year	Average yield per acre	Average protein content	Average protein per acre
	Bushels*	Pounds	Pounds
1921.....	8.5	15.02	77
1922.....	14.1	12.00	102
1923.....	7.1	13.29	57
1924.....	15.5†	11.33	105

\*Data from United States Department of Agriculture.

†Preliminary figure.

The yield of protein per acre would be quite important in a feed crop, but wheat is used principally for the manufacture of flour. The miller is interested particularly in the percentage of protein in the grain rather than the total quantity produced, as flour must contain a sufficient percentage of protein or gluten to give proper baking strength. The data on pounds of protein produced per acre, however, are important as they suggest that low-protein wheat in cool seasons may be avoided by having a proper supply of nitrates available in the soil. Under present conditions, however, the variation of the mean daily temperature of June and July is probably not only the most important climatic factor but the most important of all factors in determining the average protein content of North Dakota wheat.

While low-protein content of wheat in cool seasons may be due to lack of available nitrogen in the soil, we must not overlook the fact that extremely high temperature and hot winds, such as occurred in 1921, probably have a specific effect upon the development of the wheat berry. A combination of high temperature and low rainfall evidently produces high average protein content.



### Can Protein Content of Wheat be Increased?

The seasonal variation in climatic conditions is the principal cause of variation in protein content of crops from season to season. While the climate is certainly not under the control of the wheat grower, it is sometimes possible to control the effect of the climate.

Previous investigations at different experiment stations in this country and in Europe show that the protein content of wheat can be increased by the application of readily available nitrogen at the proper time.

The fact that low protein content is accompanied by a high yield per acre suggests that the cause of low protein may be the lack of a proper supply of available nitrates in the soil; and this possibility is further suggested by the fact that samples of high-protein wheat are found in years when the average of the crop is quite low. Wheat following sweet clover usually contains a high percentage of protein.

#### Data from Variety Plots at Fargo

Two series of the variety plots at Fargo in 1924 were seeded on sweet clover ground, while a third series was seeded on soy bean ground. Eight varieties on sweet clover land averaged 14.47% protein, while the same varieties on soy bean land averaged only 11.32%.

The average protein content of wheat following soy beans is practically the same as the average of all samples collected in Cass County in 1924—11.41%. This indicates that soy beans as a preceding crop neither increase nor decrease protein content.

TABLE IV  
EFFECT OF PRECEDING CROP ON PROTEIN CONTENT, SWEET CLOVER VS. SOY BEANS  
Wheat from Fargo variety plots

Variety	Protein	
	Series 2 after sweet clover	Series 3 after soy beans
	Per cent	Per cent
Marquis .....	14.42	10.02
Kota .....	15.87	10.88
Powers Flfe.....	15.17	10.01
Ruby .....	15.05	11.17
Red Bobs.....	13.47	9.62
Preston .....	15.64	13.78
Quality .....	11.43	14.69
Hybrid 1658.....	14.67	10.40
Average .....	14.47	11.32

An extensive use of such legume crops as sweet clover in cropping systems will probably increase the protein content of North Dakota wheat. The use of these crops will enable a wheat grower to produce

a high-protein crop in a year of low average protein content. As the trend of farming in North Dakota in the last few years has been toward diversified farming and the consequent production of forage crops and pasture for live stock, we may expect continual improvement in soil fertility. Production of a considerable acreage of wheat on sweet clover land would probably assure a limited supply of high-protein wheat in years when climatic conditions are not favorable for the production of high-protein wheat.

#### Relation of Gluten Quality and Climatic Variation

It is generally recognized that quality of gluten as a factor in baking strength is equal in importance to the quantity of protein or gluten present. Data secured by the North Dakota Experiment Station show that, in general, the baking quality of hard red spring wheat crops will vary less than the protein content. The comparatively smaller seasonal variation in baking strength may be attributed to variation in gluten quality. Climatic conditions which produce wheat of very high protein content evidently do not produce gluten of high quality. The low protein content of some crops may often be partially compensated by the high quality of the gluten. While the average protein content of North Dakota wheat has varied from 11.33 to 15.02% in the last four years, the baking quality of flour produced from North Dakota wheat has fortunately shown comparatively less variation. Owing to the compensating variation in gluten quality, the seasonal variation in quality of hard spring wheat flour is much less than the variation in protein content indicates.

#### Summary

1. The average protein content of North Dakota spring wheat crop shows considerable variation for the past four years.
2. High temperatures in June and July characterized 1921 and 1923 when protein content was high, while the low-protein years of 1922 and 1924 were characterized by subnormal temperatures.
3. Subnormal temperatures increase the length of the growing period by delaying maturity, and a long growing season is associated with subnormal temperature and low average protein content.
4. Variation in rainfall was not an important factor in determining protein content of the crops of 1921 to 1924.
5. Low temperatures in June and July are associated with high yields per acre, and the yield of protein in pounds per acre was higher in 1922 and 1924 than in 1921 and 1923.

6. High-protein wheat can be produced when climatic conditions favor the production of low-protein wheat. Preceding crops affect the protein content of wheat; and sweet clover as a preceding crop increases the protein content of wheat.

7. Baking quality of flour from North Dakota wheat shows comparatively less seasonal variation than protein content, owing to the compensating variation in quality of gluten.

#### Literature Cited

Greaves, J. E., and Carter, E. G.

1923. The influence of irrigation water on the composition of grains and the relationship to nutrition. *J. Biol. Chem.*, Vol. 58, pp. 107-112.

Le Clerc, J. A.

1910. Tri-local experiments on the influence of environment on the composition of wheat. *U. S. Dept. Agr., Bur. Chem. Bul.* 128.

Shutt, F. T.

1910. Wheat. The composition of the grain as influenced by the soil moisture content. *Cent. Exp. Farm (Canada) Report* 1908-9, pp. 140-144.

Stewart, J. F., and Hirst, C. T.

1913. The chemical, milling, and baking value of Utah wheats. *Utah Agr. Exp. Sta. Bul.* 125.

Thatcher, R. W.

1913. The chemical composition of wheat. *Wash. Agr. Exp. Sta. Bul.* 111.

## EFFECT OF HYDROGEN PEROXIDE ON RELATIVE VISCOSITY MEASUREMENTS OF WHEAT AND FLOUR SUSPENSIONS

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(Read at the convention June 5, 1925)

Reviews of literature on wheat and flour investigation fail to include results of much practical investigation which are found in United States patents, but which, for mercenary reasons, are not published. These patents, while not always scientifically accurate, are nevertheless a source of valuable information. Thus, there are patented processes and bread improvers which influence hydrogen-ion concentration, buffer value, diastatic and other enzymic activity, yeast activity, and those introducing halogenated compounds and vegetable gums. In addition to these, there is a class of improvers which has been very little investigated or at least on which very few published data are

available. This class includes substances classified as oxidizing agents. Those commonly used are peroxides, perozone, and other per-salts.

Sasse (1918) found that absorption of flour could be increased 2 to 2.5% if the wheat from which it was milled had previously been treated with 0.139% of hydrogen peroxide (3%). Higher amounts of dry gluten were obtained from treated samples. Patterson and McLaren (1918) found that it was possible to alter the percentage of ammonia and amino nitrogen in flour upon hydrolysis and thereby alter the baking strength. This they accomplished by treating flour with 1½% solution of hydrogen peroxide for 8 hours. Total nitrogen remained the same, but ammonia nitrogen increased 5.7%, while amino nitrogen decreased 5.8%. The authors suggest this may be due to oxidation of amino compounds to ketonic acid and ammonia. Patterson (1918) stated that 0.033% hydrogen peroxide added to dough results in increased absorption.

As no satisfactory explanation has been given for improved baking qualities due to oxidation, this investigation was undertaken. The results of the work of Sasse and of Patterson, as stated before, suggest increased hydration capacity of flour. This is, however, not the only object in using oxidizing agents, for such agents are claimed to have an effect on loaf volume and texture which can not be attributed to additional absorption. The first aim of this investigation was to determine, if possible, the extent of increase in absorption, measured as hydration capacity, by means of relative viscosity of flour or wheat in water suspensions.

Considerable preliminary investigation brought out the fact that in making viscosity measurements on flour and water suspensions when electrolytes were not removed, time of suspension was an important factor. Time required for reaching maximum hydration varied with different flours, and as to determine maximum hydration capacity would require not less than six determinations on a single flour; and as viscosity when mixed and run at once was for most flours proportionately lower than maximum viscosity reached on standing, it was concluded that for purposes of comparison and for practicability the shortest or "no-time" method could be used. The method worked out for measuring viscosity of flour and water suspensions was as follows:

Mix in a 250-cc. beaker by means of a short spatula and in such a manner that all lumps are broken up, but at the same time causing as little foaming as possible, 20 grams flour (13.5% moisture basis) and 100 cc. distilled water. Add to this 2 cc. concentrated lactic acid and stir for about 30 seconds. Pour into large bowl of MacMichael viscosimeter and take reading, using disc bob, No. 30 wire, and rotating



bowl 20 R. P. M. This reading will be spoken of in this paper as viscosity with lactic acid or, more simply, with acid. To this same suspension is then added one drop of 3% hydrogen peroxide, and after stirring another reading is taken as before. The difference between this reading and that with lactic acid will be spoken of here as the increase with peroxide. There is no doubt that other peroxides not introducing depressing electrolytes might be used in place of hydrogen peroxide. Considerable difficulty was experienced in taking readings if peroxide was added to a suspension that had not been previously acidulated. Whether or not the viscosity as determined with acid in this work is of any significance may be questioned. In tables presented in this paper such viscosity measurements are recorded for information only.

A great many difficulties present themselves when one undertakes to make viscosity measurements of wheat meal. First, there is the method of grinding samples. A simple experiment on effect of size of flour particles on viscosity disclosed the fact that the finer the middlings are ground, the higher the viscosity. This experiment was performed on first middlings in a large mill and results were as follows:

	Viscosity determined at once °MacM	Viscosity after 1 hour suspension °MacM
Middlings coming to roll.....	140	173
Ground very coarse.....	183	243
Ground as usual.....	235	300
Ground very fine.....	255	322

This seemed sufficient reason to believe that in order to secure consistent results the endosperm must in every case be reduced to particles of equal size. As the inner portion of soft wheat is more likely to be reduced to a relatively fine state with ease, in fact, so easily that it is almost impossible to grind such wheat without reducing a considerable portion to flour, it seemed only logical to conclude that all samples should be reduced to that fineness attained in grinding the softest wheat. It was also found that the extent to which bran was ground affected its absorptive properties. Thus a sample of clean broad bran prepared by breaking dry wheat on corrugated rolls, absorbed roughly 29% of its weight of water. Another portion of the same bran was ground twice through an Enterprise coffee grinder and its absorption reduced to 17%. While the method finally decided upon for measuring viscosity of wheat meal in water involves two determinations, one with acid and one without, thus arriving at natural hydration of bran and flour, it is apparent that an error may still creep in if bran is not uniformly pulverized.

Taking all these possible sources of error into consideration, the following method was worked out for grinding wheat samples: Grind wheat through Enterprise coffee grinder adjusted to crush without producing much flour. Sift on experimental mill sifter clothed with 34 wire and 10 xx flour cloth. Grind the overs of 34 wire through coffee grinder, sift as before, and again grind and sift overs of 34 wire. This makes in all three grindings through the coffee mill. A small amount of endosperm still adheres to the bran. Overs of both 34 wire and 10 xx are ground through smooth rolls and sifted as before. Overs of 34 wire are now removed, but retained and overs of 10 xx reground and rebolted until free from endosperm. All bran and flour is then thoroly mixed and the sample is ready for viscosity determination.

The no-time method as used on flour was not applicable for viscosity measurements on a suspension of wheat meal and water. Successive readings taken one immediately after the other show a gradual decline over a period of several minutes. Apparently the electrolytes are slowly entering into the reaction and not until their full effect is reached is one able to secure two readings that check. This method, which is in some respects similar to that published by Morgan (1924), was found to give the best results. Two suspensions are made up using 30 grams wheat meal (13.5% moisture basis), and 100 cc. distilled water. To one is added 2 cc. concentrated lactic acid. Both are allowed to stand for one hour at 25° C. with occasional stirring. Readings are then made as with flour and one drop of hydrogen peroxide is added to the suspension containing acid, after which another reading is made. Therefore, on wheat we have 4 results: without acid (A), with acid (B), difference between (A) and (B), and the increase with peroxide over (B).

As stated above, part of the original investigation was outlined to determine whether or not peroxide increased hydration capacity as measured by viscosity methods. That viscosity was increased by peroxide was very easily proved, and the investigation soon resolved itself into a study of variations in increase due to flour grade and wheat type.

Through the courtesy of chemists in different sections, various types of wheat were collected and a study was made of viscosity by the method outlined. The results are given in Table I. That there is no correlation between protein content and viscosity with either acid or peroxide, is quite apparent. There does, however, seem to be a relation between relative hardness of the wheat and increase with peroxide. Relative hardness of these types can not be expressed in

numerical values, as it may only be arrived at from observation of wheat and its behavior and appearance during grinding. While no conclusive data can be presented, practical experience has led to the conclusion that high ash content in flour results when wheat showing

TABLE I  
INCREASE IN VISCOSITY OF WHEAT MEAL IN WATER SUSPENSIONS UPON THE ADDITION OF  
HYDROGEN PEROXIDE

Identification of sample	(All results reported on 13.5% moisture basis)			Ash
	Weight per bushel	Crude protein (N×5.7)		
	Pounds	Per cent		Per cent
Idaho Northern Spring Marquis.....	59.6	13.90		1.663
Kansas Dark Hard blended.....	58.0	13.72		.....
Idaho Turkey.....	60.9	13.69		1.556
Black Hull.....	.....	13.43		1.891
Texas .....	.....	12.75		1.624
Idaho Hard White Baart.....	61.1	12.64		1.749
Idaho Soft White Club.....	58.6	12.50		1.530
Pacific Coast blend of Turkey Red and Baart .....	62.0	12.45		1.672
Kansas 100% Dark Hard.....	57.8	12.15		1.966
Kansas Hard blended.....	59.7	12.00		1.838
Idaho Turkey.....	61.4	11.97		1.479
Kansas 100% Dark Hard.....	58.1	11.96		1.801
Texas .....	.....	11.90		1.733
Minnesota Northern Spring.....	60.4	11.63		1.502
Missouri Soft.....	59.7	11.19		1.818
Kanred .....	.....	11.06		2.032
Michigan Soft.....	61.2	10.98		1.655
Kansas Ordinary.....	61.2	10.75		1.747
Kansas Ordinary.....	60.0	10.15		1.812
Idaho Soft White Dicklow.....	60.4	9.48		1.615
	Viscosity without acid (A) °MacM	Viscosity with acid (B) °MacM	B-A °MacM	Increase over B with H <sub>2</sub> O <sub>2</sub> °MacM
Idaho Northern Spring Marquis	93	177	84	145
Kansas Dark Hard blended....	122	206	84	182
Idaho Turkey.....	121	235	114	737
Black Hull.....	.....	116	.....	84
Texas .....	114	198	84	205
Idaho Hard White Baart.....	72	115	43	113
Idaho Soft White Club.....	61	93	32	45
Pacific Coast blend of Turkey Red and Baart.....	80	146	66	189
Kansas 100% Dark Hard.....	139	245	106	1400
Kansas Hard blended.....	77	135	58	110
Idaho Turkey.....	89	174	85	174
Kansas 100% Dark Hard.....	121	205	84	955
Texas .....	99	156	67	147
Minnesota Northern Spring....	73	141	68	83
Missouri Soft.....	70	99	19	40
Kanred .....	.....	104	.....	116
Michigan Soft.....	67	94	27	25
Kansas Ordinary.....	69	124	55	69
Kansas Ordinary.....	78	122	44	71
Idaho Soft White Dicklow.....	45	62	17	35

high increase with peroxide is milled. Flour also has a low viscosity with acid but a large increase with peroxide, altho wheat may have had high viscosity with both acid and peroxide. Ash content of wheat was determined and included in Table I. Altho high-ash flour results from milling wheat showing large increase in viscosity with peroxide, no correlation is noted between ash content of wheat and increase with peroxide. This, however, is not significant, as ash in wheat is no criterion of ash in flour milled from it.

In Table II are recorded the results of analysis and viscosity determinations of mill streams of a typical mill grinding Kansas Hard Winter wheat containing 12% protein. It will be observed that in general the middlings flour shows greatest increase with peroxide while break flour and lower grades show less increase. Moisture content as recorded in these tables was determined by drying in an electric oven at atmospheric pressure and 104° C. for six hours.

TABLE II  
INCREASE IN VISCOSITY OF FLOUR AND WATER SUSPENSIONS OF MILL STREAMS UPON  
THE ADDITION OF  $H_2O_2$ \*

(Viscosity results only are reported on 13.5% moisture basis)					
Identification of sample	Moisture	Ash	Crude protein (N×5.7)	Viscosity with acid	Increase over acid with $H_2O_2$
	Per cent	Per cent	Per cent	°MacM	°MacM
Patent .....	13.42	.351	10.40	286	229
Clear .....	13.38	.693	12.56	167	93
First middlings.....	12.75	.310	9.92	284	146
Second middlings.....	12.80	.303	10.04	324	443
Third middlings.....	12.45	.310	10.16	335	413
Fourth middlings.....	12.85	.376	10.56	261	184
Fifth middlings.....	12.40	.430	10.92	268	152
Sixth middlings.....	12.08	.486	11.40	281	288
Seventh middlings.....	12.55	.940	11.40	49	6
First sizings.....	13.30	.386	9.96	254	60
Second sizings.....	13.30	.390	9.60	211	52
Third sizings.....	13.00	.506	11.52	202	59
First dust.....	12.70	.330	10.16	272	198
Second dust.....	12.50	.456	11.36	261	219
Third dust.....	12.60	.686	12.20	209	231
First break.....	13.70	.610	10.48	121	64
Second break.....	13.65	.493	10.72	182	68
Third break.....	13.80	.466	11.56	214	66
Fourth break.....	13.30	.526	12.28	205	65
Fifth break.....	12.90	.846	12.92	56	53
Purifier and elevator head suction.....	12.65	.553	11.10	193	72
Break roll suction.....	13.20	.735	13.77	175	91
Bran duster flour.....	12.80	.940	12.84	35	45
Shorts duster flour.....	12.05	1.146	12.44	43	44

\*This flour was milled from wheat designated in Table I as Kansas Hard blended.



In order to ascertain if peroxide acted on gluten and produced increased viscosity, a sample of hard wheat short patent flour was washed to remove the electrolytes. A total of 1500 cc. of water was used in washing 20 grams of flour which was finally made up to 100 cc. As shown in Table III, only slight increase was produced with peroxide. The washing process required 2 hours. In order to be sure that the substance acted on had not been denatured by contact with water, a 20-gram sample of the same flour was suspended for 2 hours in 100 cc. distilled water, and the viscosity was measured. These results are also recorded in Table III. From these results it was concluded that peroxide acts not on the gluten, but on some water-soluble constituent. This conclusion was supported by the following experiment. A short patent flour containing 0.37% ash and 11.70% protein and showing an increase with peroxide of 350° MacMichael was employed. A suspension of 200 grams flour and 800 cc. distilled water was allowed to stand for one hour with occasional stirring. An additional half hour was allowed for settling, after which the supernatant liquid was decanted off and filtered with suction through an asbestos pad in Gooch crucible. One drop of 3% hydrogen peroxide added to 100 cc. of the clear extract produced a gel that apparently took up all the water. Owing to extreme difficulty encountered in trying to filter and wash the gel, attempts to purify it have been unsuccessful.

TABLE III

EFFECT OF REMOVAL OF ELECTROLYTES AND OTHER SOLUBLE MATERIAL ON INCREASE IN VISCOSITY WITH HYDROGEN PEROXIDE

	Viscosity with lactic acid °MacM	Increase in viscosity with H <sub>2</sub> O <sub>2</sub> °MacM
No-time method.....	255	132
Suspension set 2 hours.....	368	406
After washing out soluble matter.....	1038	12

Morgan (1924) reports low viscosity in flour dusted from the germ section and suggests that "wheat oil" may have a detrimental effect. To throw possible light on identification and location of the substance acted on by peroxide and to ascertain if the germ portion of wheat berry showed a lower viscosity, grains of wheat were carefully cut in two and divided into germ end and tip end. Having shown that peroxide acted upon a soluble substance, a sample of wheat was selected for this work that showed no evidence of having been rained upon, or otherwise wetted, thus precluding any possibility of redistribution of soluble constituents in the berry. Analysis and viscosity of the whole wheat and of the two ends are shown in Table IV. It will be noted that the germ portion has the highest ash and protein

content but the lowest viscosity with acid and peroxide. This fact together with results shown in Table II wherein middlings flour was shown to have the greatest increase with peroxide indicates that tempering wheat does not redistribute in the berry that substance acted on by peroxide.

TABLE IV  
EFFECT OF  $H_2O_2$  ON VISCOSITY OF WHOLE WHEAT MEAL AND ON GERM END AND TIP END  
(All results reported on 13.5% moisture basis)

	Crude protein (N $\times$ 5.7)	Ash	Viscosity without acid (A)	Viscosity with acid (B)	(B)-(A)	Increase over (B) with $H_2O_2$
	Per cent	Per cent	$^{\circ}$ MacM	$^{\circ}$ MacM	$^{\circ}$ MacM	
Whole wheat meal..	12.15	1.97	139	245	106	1400
Germ end.....	12.34	2.08	105	217	112	204
Tip end.....	12.03	1.91	228	299	71	*

\*Too high to read.

In our laboratory we have never been able to duplicate with the experimental mill results obtained on the large mill, especially with respect to increased swelling with peroxide. Experimentally milled flour always shows the least increase. Comparatively low readings with peroxide are usually obtained on flour produced in small or inefficient mills. Imperfect separation of various stocks is the most logical explanation of this problem, altho another possible solution that has not been investigated is temperature of stocks after grinding.

As before stated the purpose at the outset of this investigation was to determine, if possible, the cause of improved baking results when oxidizing agents are employed in the dough batch. We are yet far from the answer. Further investigation is being conducted on doughs. Some preliminary results indicate that swelling with peroxide is decreased during fermentation.

### Conclusions

1. Hydrogen peroxide produces increased hydration capacity of flour or wheat meal and water suspensions as determined by viscosity with MacMichael viscosimeter.
2. Different types of wheat vary greatly in their behavior toward peroxide. Excluding Durum wheat, on which very little work was done, there seems to be a relation between the hardness of wheat and its increase in viscosity with peroxide.
3. Middlings flour shows greater increase in viscosity with peroxide than do lower grades.
4. The substance effected by peroxide is soluble in water.
5. Inferior milling produces flour which shows low increase in viscosity with peroxide.

### Literature Cited

- Morgan, R. W.  
1924. A practical application of the viscosimeter to the mill. *Cereal Chem.*, Vol. I, pp. 288-292.
- Patterson, C. J.  
1921. Process of bread-making. U. S. Patent, 1385842.
- Patterson, C. J., and McLaren, L. M.  
1918. Protein hydrolysis. *Jour. Am. Assn. Cereal Chem.*, Vol. 3, pp. 10-16.
- Sasse, A. R.  
1918. Investigation of peroxide process. *Jour. Am. Assn. Cereal Chem.*, Vol. 3, pp. 20-22.

## A STUDY OF METHODS OF DETERMINING THE LOAF VOLUME OF BREAD

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(Read at the convention June 2, 1925)

### Introduction

Loaf volume of bread, as determined in the experimental baking laboratory, is one of the most important expressions of the strength of flour, yet it is one of the measurements used which is most variable and of which there seems to be no generally accepted method. In order for a method of determining loaf volume of bread to be practical it must meet the following requirements:

1. It must be reasonably rapid.
2. It must be accurate and capable of being checked frequently for accuracy.
3. It must have a range, making it possible to measure both large and small loaves.
4. It must not impair the loaf for other determinations.
5. It should be one that can be uniformly applied in all experimental laboratories.

A review of the methods employed in measuring bread indicates that few of them meet these requirements. Some methods are highly accurate but very difficult in application; others are easily and quickly applied but are highly inaccurate and do not afford any convenient means of checking the accuracy.

The principle of the various methods of measuring loaf volume of bread may be grouped under three heads:

1. Using the circumference of the loaf as an index of the relative size of loaves.
2. Determining the actual volume of the loaves by displacement of seed.
3. Determining the volume of the loaves by the displacement of a liquid.

The first of these may serve the purpose of comparing loaves in any one laboratory but can hardly be used for comparing loaves in one laboratory with those in another. Obtaining the volume by displacement of seed is by far the most easily applied of any of these three methods, but it must be used with caution. Measuring the loaf volume by the displacement of liquids is an accurate method, but has its limitations because it is necessary to coat the loaf with some material to prevent the entrance of the liquid.

The present study was undertaken with a view to comparing the efficiency of the methods more commonly employed in measuring bread. Most of the tests were made by the displacement of seed, as this is the principle most commonly employed and the one for which equipment was available. Comparisons were made of four different kinds of measuring devices and six different kinds of seeds. The volume by displacement of water was determined on one loaf.

#### Method Employed

**Standard loaves.**—Standard loaves of varying sizes and shapes were fashioned from wood and well enameled to make them impervious to water. Three types of loaves were represented:

1. A tall narrow loaf as baked in a pan  $6\frac{1}{2}$  inches long, 3 inches wide, and  $5\frac{1}{2}$  inches deep.
2. A tall round loaf as baked in a pan  $4\frac{1}{2}$  inches in diameter and 8 inches deep.
3. A low long loaf as baked in a pan 9 inches long,  $3\frac{1}{2}$  inches wide, and  $3\frac{3}{4}$  inches deep.

These loaves were carefully measured by the displacement of water in a specially constructed container. By this method it was possible to check the volume of the various loaves within one cubic centimeter for two or more determinations. This insured the accuracy of the standard loaves used in this study.

Some difficulty was experienced in finding a container sufficiently accurate to measure the standard loaves. At first an open vessel was used, but it was found inaccurate and could not be easily checked. The container finally used consisted of a strong wooden box of 4075 cubic centimeters at 20° C. The cover of this box was clamped on



with bolts and the joints were fitted with thick rubber packing. Two glass tubes  $\frac{1}{2}$  inch in diameter were fitted into opposite corners of the cover in such a way as to serve as standpipes. These tubes served the double purpose of furnishing a means of filling the box with water and exhausting the air and also reading the water to the nearest cc. by means of graduations. At the time of reading, the temperature of the water was determined by inserting a thermometer through these tubes. The water was weighed to the nearest tenth of a gram as it was poured into the box. The temperature of the room was maintained at 20° C. by means of an electric fan which made it possible to keep the temperature of the water constant.

The three standard loaves used had the following volumes:

Tall narrow loaf.....1995 cc.

Tall round loaf.....1645 cc.

Low long loaf.....1660 cc.

**Measuring devices.**—Five devices were used for measuring loaf volume by displacement of seed or liquid. These are described as follows:

Name and principle of measuring	Amount of seed required cc.	Read to nearest cc.
Hour-glass—Standard loaf used, excess seed deposited in tube through hour-glass arrangement. Volume read directly on tube.....	2400	10
Kansas—Volume of container known. Seed not displaced by loaf is poured into graduated tube. Volume of loaf is the difference.....	3120	10
Montana—Seed used fills box, and all seed displaced by loaf is deposited in graduated tube.....	10450	25
Weighing—Volume of container known. Seed not displaced by loaf is weighed and its volume computed from known unit weight. Volume of loaf is computed by the difference .....	5100	1
Displacement by liquid—Loaf is coated with water-proofing material and submerged in vessel of water, and water displaced is measured.....	.....	1

These measuring devices are named, for convenience, from the principle under which they operate and from the place where they were originated or used. The hour-glass method is the one most commonly used and is found in many laboratories. The Kansas method was first limited to the measuring of tall round loaves, but has later been modified to measure loaves of various shapes. The Montana method was devised at the Montana Experiment Station about ten years ago, but has not been used since 1922, when the hour-glass method was adopted. The weighing method was suggested by a German investigator and has been occasionally tried in this country. The two water-proofing materials used are paraffin and a solution of rubber in benzine.

### Results Obtained

The results obtained in this brief study of this most interesting subject are summarized in the three tables appended. It is not the intention of the author that these data be accepted as final, but that they be used merely to indicate what may be expected when a loaf of bread is measured by one of these methods.

The results obtained are discussed under different kinds of measuring devices and different kinds of seeds.

**Kinds of measuring devices.**—The data obtained with flaxseed in the four different measuring devices are given in Table I. The three types of standard loaves were measured. The tall narrow loaf with a standard volume of 1995 cc. showed a volume of 1870 with the hour-glass method, 1940 with the weighing, and 2100 with the Montana method. The tall round loaf with a standard volume of 1645 cc. showed a volume of 1710 with the Kansas method, and 1625 with the Montana. In nearly all cases the deviation from the standard volume is more than one would expect.

TABLE I

COMPARISON OF VOLUMES OF STANDARD LOAVES AS DETERMINED BY DIFFERENT MEASURING DEVICES (FLAXSEED USED)

Measuring device	Description of loaf	Standard volume of loaf	Volume as measured	Deviation from standard
		cc.	cc.	cc.
Hour-glass	Tall narrow	1995	1870	-125
Hour-glass	Low long	1660	1600	- 60
Kansas	Tall round	1645	1710	+ 65
Kansas	Low long	1660	1720	+ 60
Montana	Tall narrow	1995	2100	+105
Montana	Tall round	1645	1625	- 20
Weighing	Tall narrow	1995	1940	- 55

When submerged in an open vessel of water the tall narrow loaf, which had a standard volume of 1995 cc. showed a volume of 1960 cc.

**Kinds of seeds.**—A comparison of the volumes obtained on the tall narrow loaf with a volume of 1995 cc. and the low long loaf with a volume of 1660 cc. with the different kinds of seed in the hour-glass measuring device is made in Table II. The deviations from the standards are minus in all cases. Millet seed seemed to give the most nearly correct result for the tall loaf, while flaxseed gave the best result for the low loaf.

TABLE II

COMPARISON OF STANDARD LOAVES AS DETERMINED BY DISPLACEMENT OF DIFFERENT KINDS OF SEED (HOOR-GLASS MEASURING DEVICE)

Kind of seed	Description of loaf	Standard volume of loaf	Volume as measured	Deviation from standard
		cc.	cc.	cc.
Rape	Tall narrow	1995	1850	-145
Rape	Low long	1660	1590	- 70
Flaxseed	Tall narrow	1995	1870	-125
Flaxseed	Low long	1660	1660	- 60
Sweet clover	Tall narrow	1995	1860	-135
Millet	Tall narrow	1995	1920	- 75
Vetch	Tall narrow	1995	1990	- 95
Wheat	Tall narrow	1995	1870	-125

The range in readings obtained together with the deviations from the standard for tests with different kinds of seeds in the Kansas measuring device is given in Table III. Millet and peas showed the greatest variation in range of three or more readings while peas showed the greatest deviation from the standard. Millet and sweet clover showed the least deviation from the standard.

TABLE III

COMPARISON OF LOAF VOLUME AS DETERMINED BY DISPLACEMENT OF DIFFERENT KINDS OF SEEDS (KANSAS MEASURING DEVICE. TALL ROUND LOAF 1645 CC. VOLUME)

Kind of seed	Average volume of loaf	Range in volume for three or more tests	Deviation from standard of 1645 cc.
	cc.	cc.	cc.
Rape .....	1690	0	+ 45
Flaxseed .....	1710	0	+ 65
Sweet clover.....	1675	10	+ 30
Millet .....	1645	30	0
Vetch .....	1700	0	+ 55
Wheat .....	1720	0	+ 75
Peas .....	1800	30	+155

### Conclusions

It is not the intention of the writer to draw definite conclusions from this brief study or to recommend any one method of testing over others, but merely to point out certain observations in the study.

1. A greater variation from the standard loaf is found than might be expected.

2. It is not practical to measure loaf volume closer than 10 cc.

3. A greater deviation from the standard was observed by the use of different devices than by the use of different kinds of seeds in any one device.

4. Certain measuring devices like the Kansas give results consistently higher than the standard, while other devices like the hour glass give results consistently lower than the standard.

5. A method of measuring loaf volume which has a small range in a number of readings is to be preferred even though the deviation from the standard may be great. This makes it possible to apply a correction.

6. It is desirable that a standard loaf of known volume be used as a comparison with all systems of measuring. This standard should be of the same shape and much the same size as the loaves of bread tested.

### THE BAKING TEST

By E. E. WERNER

Consulting Engineer, St. Louis, Mo.

(Read at the convention June 2, 1925)

For the last twenty or more years my work has been intimately connected with flour bleaching and treating. During the early days of this work fantastic ideas prevailed as to the detriment to mankind due to bleaching. Investigators both in this country and abroad claimed to find inferior baking quality due to it. A recognized or even semi-scientific baking test did not exist, and I may say, with deference to all, does not exist now. It became of interest to owners of the patents to verify whether or not bleaching in fact injured the baking quality of flour, and I was called upon to give thought to the problem. The difficulty which confronted me will be as apparent to you now as it was to me at the time. Baking quality is purely relative, as it so largely depends upon the quantity of yeast, the mineral content of water, the amount of salt, and above all, the idiosyncrasies and the dexterity and care of the baker; also upon his notions as to added material in general and the physical conditions under which he operates. In this modern day it also depends upon and is complicated by the flour's resistivity, or if it pleases some of my friends better, to its response to the haphazard additions of various mixtures or chemicals tending to improve baking quality in some respects. Over-emphasis was placed then, as now, on the factor of absorption as having direct relation to baking quality, a heresy in my opinion. Flour having an excellent yield in terms of bread may nevertheless be deficient in quality. But disregarding my opinion, we all know that a material variation in absorption will materially alter the resultant character of the bread. I will not arrogate myself to say which is



better bread, but I know two eminently successful baking organizations who buy the same flour from the same mill, one producing bread at a commercial net absorption of 54% and the other of 62%. The flour itself seems suitable to both. I think that the attempt to judge baking quality from the standpoint of gluten quality was and still is hardly worth while, considering the enormous amount of work which has been and still is being done in this direction by investigators of whom the earlier have been disproved or have recanted in part or in toto, and the later ones are still in the domain of hypothesis or at the best, theory. Baking quality appears to be more a manifestation of chemical and physical environment in the process of baking than an inherent quality of the nitrogenous or other matter in flour. I believe that Dr. Swanson was quite right when he stated in his recent paper before the A. O. M. that "quality is a relative term and cannot be discussed without some reference to the intended use of the wheat." May I add to this, "and of the flour." I hold, with him, that a high quantitative gluten factor is not necessarily an indication of good quality from the baker's standpoint, altho it is a fairly reliable guide of the baking quality of a flour in the absence of the baking test.

However, I have drifted. It became desirable and necessary to ascertain by comparative baking what, if any, difference was due to bleaching. It was recognized that a baking test to be of value in furnishing data for interpretation must, when performed in duplicate or in triplicate, give concordant results within a small reasonable factor of error. Dr. A. Mauritizio had published in 1903 his work "*Getreide. Mehl und Brot*," which fortunately supplied foundation for such a test. With considerable modification it was then adopted and used. The results obtained, while by no means perfect, were and have been of great value. The test consists of baking in duplicate a quantum of flour under conditions as nearly as possible maintained uniform. The Freas incubator is used for fermentation and final pan proof and the Freas high-temperature oven for baking. The thermostatic control is well within plus and minus one degree centigrade, and to assure uniform exposure the baking oven contains a motor driven shelf revolving at about 20 R. P. M. I do not intend to bore you with the details of the test, as after all they are subject to variations and are merely routine. I will be delighted to give the work step by step to your baking committee or to anyone who cares for the details. For lack of time, I will now say only that the dough is given a first and second rise and a period of recovery prior to molding, and that the handling is quite in accord with ordinary bakeshop practice. As practiced, the test dough is made in duplicate from 100 grams of

flour, 3 grams of yeast,  $2\frac{1}{2}$  grams of sugar, and 1 gram of salt, and with an absorption norm of 60%, with flour moisture content norm of 12.5%. Correction is made for lower moisture in flour at the rate of  $1\frac{1}{2}$  to 1. The test calls for care but is not laborious. After some training several skilled and unskilled operators have been able to each produce eight duplicates or sixteen singles in a working day of eight hours. The test permits of and includes a careful check of losses at each step and throughout the entire operation. The theoretical weight of all material used for each single bake is 166.5 grams at normal absorption. At the time of panning, after being handled five times *without the use of dusting flour*, the total losses against theoretical weight are well within  $6\frac{1}{2}$  grams, in other words, approximately  $3\frac{3}{4}\%$ , which compares not unfavorably with the invisible losses of the bakeshop. The difference between duplicates will not exceed  $\frac{1}{2}$  gram, to-wit:  $\frac{1}{2}$  of 1 per cent. The total loss from theoretical weight to bread one hour after baking approximates 16%, giving a total loss in the oven of  $12\frac{1}{4}\%$ . This again compares not unfavorably with standard bakeshop practice. For several years I have been able to check the losses in baking and fermentation in a fairly large bakeshop. At random I select the month of August, 1921. During this month there was a total production of 665,850 pounds of one kind of bread. Measurements were made daily on a number of loaves supplied by the foreman. The actual bakeshop losses from dividing machine to nine hours after baking were 11.5%, and the invisible losses during any of the months greater than the ones of the laboratory test. The loaves are measured volumetrically by displacement with sized mustard seed and the apparatus for this is accurate enough to give duplicate and triplicate measurements well within 1%. The duplicate loaves are practically identical in external and internal characteristics. Flours from the same mill made from approximately the same wheat blend can be easily identified although from different shipments.

As you see, there is nothing novel about the method. It merely reduces the baking test to a more or less precision operation, bringing the limit of error between duplicates to a known factor not much greater than the errors of other tests. These bakes furnish ample evidence for a priori reasoning and in my opinion the baking quality of flour as yet does not render itself to reasoning a priori from any factor or factors.

A number of trivial objections and one serious objection to the test have been made. Let me dismiss the trivial before discussing the other. "The quantity of flour used is too small." I find this a comfortable quantity to work with. The feel of the entire mass is

within the compass of one hand. This facilitates the kneading of duplicates, one in each hand, and permits reversal of the hands to compensate for dexterity. Cutting the loaf lengthwise will show crumb color and texture in a slice equal in size to ordinary commercial bread. These small quantities can be weighed more accurately than larger ones with the equipment available to the ordinary worker. I weigh the flour to plus and minus  $1/10$  gram, the remainder of the solid material to plus and minus 5 milligrams in an ordinary torsion or apothecary balance, and I measure the water with pipette and burette. The mass is after all only a matter of degree. One hundred grams are not much more remote from 400 grams than are 400 grams from the 450,000 grams customarily handled in the bakeshop. The quantitative relation between crust and crumb is almost identical with the commercial loaf. That the losses are not unduly distorted in such a small loaf, I have spoken of before. The objection that the test occupies too much time and is too much trouble is also trivial. I have answered this. Eight duplicates or sixteen singles are readily within the possibilities of a high school girl after a few weeks training. A skilled assistant will do better.

The major objection to the test is its practice under rigidly maintained conditions of temperature and time. The prevailing modern school takes the position that the baking test must be conducted to obtain optimum results by variation of fermentation period, the sense of touch, and even by artistry in baking. The tendency appears to be to force the test into purely empirical lines without much endeavor to make it even comparative, not to speak of scientific. It is the ambition of miller as well as of baker to produce a uniform product. Daily control under maintained conditions, giving attention to accuracy and care in measurements, has in my hands been a very comprehensive control of the uniformity in milling in relation to baking. Also the lack of uniformity. Similar control in a large bakeshop has thoroly convinced me that such a baking test is of greatest assistance in maintaining a uniform product either by blending or without. I recall an incident of forty consecutive carloads of flour delivered to a large bakeshop. The delivery covered a period of several months. They baked so uniformly that the record sheets were almost duplicates. Subsequently a car or two proved to be inferior. The mill first challenged the test, altho with forty cars in their favor and only two against them. It was finally admitted that the two cars in question had been made from a different wheat blend. My records show a number of such incidents.

Needless to say that a test which renders itself to control will also render itself to constructive and creative work. The point I wish to

make is merely this. This test gives sufficient concordance to permit of reasonably sound interpretation of results each day and from day to day.

Another objection is that the ingredients used are primitive and that better loaves can be produced when adding milk, malt, fat, and other things. As I understand it, the baking test is for the purpose of ascertaining the intrinsic value of the flour for the purpose of baking. We are testing the flour and not the influence of the added material upon the flour. Of course we can do this, but that is a later and a different step. At its worst, such a test furnishes information and foundation for subsequent tests now on firmer ground. However, I have not found it difficult to foretell from the routine test conditions what a given flour might do under different conditions and with the addition of materials as a rule used in the bakeshop.

Perhaps I have been wrong, according to orthodox procedure, not to bore you with the minute details of cuts and folds, of temperature and time. If I have been wrong it can be easily remedied. Of necessity I have been vague. For this I ask your pardon. It is hard to compress years of experience into a few minutes' talk.

## THE RESIDUAL SUGAR CONTENT OF BREAD

By C. B. MORISON

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(Read at the convention June 4, 1925)

Taste and flavor are among the most important characteristics of a high quality bread. This is recognized in bread scoring systems. At the American Institute of Baking the official score for ideal bread requires 20 points for taste and 15 for flavor, which is equivalent to 50 per cent of the 70 points allotted to the internal characteristics of the loaf. This is also 35 per cent of the total score of 100 points.

In this country there seems to be a national predilection for sweets. Some bakers feel that in consequence the much desired wheat flavor of bread has been sacrificed to the popular demand for sweetness. It is often remarked that bread is becoming more like cake. Other bakers hold opposite views, and it would be difficult to secure complete agreement among the trade on this question.

Visitors from abroad have in our hearing frequently commented on the sweet taste and flavor of American bread, and in most cases note



with surprise the quantity of sugar and other ingredients used in bread formulas and the increased cost of production which follows from their use.

Thus, the relation of the amount and kind of sugar used in formulas to the production of sweetness of taste and flavor in the baked bread is of essential interest. How much sugar will be found in bread made from various formulas containing commercially pure sugar or available sugar forming ingredients? What is their qualitative and quantitative influence on the taste and flavor of the bread?

The term residual sugar content of bread is used here to denote the kind and amount of sugar present in the bread baked from a fermented dough. The residual sugar content may be dependent upon several factors, among which are the kinds and amounts of sugar originally added to the dough, sugars produced by enzymatic activity, the amount of yeast used, the temperature and time of fermentation, hydrogen-ion concentration and other factors which influence enzyme action; finally, oven conditions, especially those of temperature, and time of baking.

The ingredients commonly employed for the fermentable carbohydrate requirements of bread doughs are sucrose from cane or beet, refined dextrose (d-glucose), sold under trade names such as cerelose, malt extracts or syrups, and sweetened condensed milk. In addition to these commercially pure individual sugars, and sugar containing ingredients, various partially hydrolyzed starch products are employed containing variable amounts of reducing sugars, dextrans and soluble starch. Malt flours possessing diastatic power may also be mentioned.

There is little information in bread and baking literature on the residual sugars found in bread made from known formulas under observed or controlled fermentation conditions. It is therefore desirable to study the sugars of bread in materials whose history is known.

The results reported below were obtained from an examination of bread made according to commercial formulas, mixed, fermented and baked in the Institute Bakery by M. H. Joffe.

In our preliminary work on the residual sugar content of bread we have used cane sugar, cerelose, sweetened condensed milk and dried whole milk in sponges and straight doughs, in commercial formulas, mixed, fermented and baked under known conditions.

Determinations of sugar have been made in the bread from these doughs and the following results may be reported as typical:

A sponge and dough were prepared which contained the following sugar-carrying ingredients expressed as per cent on the basis of flour as 100:

Malt extract.....	1.67%
Cerelose .....	2.30%
Sweetened condensed milk.....	2.30%
Powdered, skimmed milk.....	2.78%

The total amount of sugar introduced into the dough (sum of total sugars, maltose, glucose, sucrose and lactose) from these ingredients was approximately 3.2% of the total weight of the mixed dough of flour, water, yeast, salt, shortening, and the above sugar-containing ingredients. The flour contained 0.75% total sugar, calculated as maltose and sucrose by copper reduction. This amount of sugar calculated from the amount of flour used increased the total sugar in the dough from approximately 3.2% to 3.9%.

The total lactose in the dough was approximately 1.00% as calculated from the determination of lactose in the skimmed milk powder and the sweetened condensed milk, leaving about 2.9% glucose, maltose and sucrose available for fermentation.

The malt extract had a low diastatic power, 2° Lintner. The diastatic power of the flour was not determined.

The total yeast used in the sponge and dough was 2% on the basis of flour as 100.

The sponge was mixed at 80° F. and fermented 190 minutes. The sponge was mixed with the dough at 82° F. and fermented for 30 minutes. It was then sent to the bench, scaled, rounded, molded by hand, panned and given a proof of 45 minutes at 92° F. The dough was then transferred to the Duhrkopf oven and baked for 25 minutes at 475° F. After cooling one hour the bread was weighed, wrapped and sent to the laboratory for examination.

The moisture content of the fresh bread was 37.18%, and of the ground sample taken for the determination of sugars was 6.25%.

The analytical difficulties connected with the identification, separation and determination of the sugars in food products such as bread are complicated.

We are not prepared at this time to recommend special analytical schemes for the residual sugars in bread. The results reported were obtained by extracting the dried ground bread with ethyl alcohol and the reducing sugars determined before and after inversion by the official method of the A. O. A. C.<sup>1</sup>

The clarification of the aqueous solution after evaporation of the alcohol by neutral lead acetate, and subsequent removal of the lead by anhydrous sodium carbonate was followed according to this method, but experiments with other clarifying agents, such as sodium tungstate, are being studied in this connection.

<sup>1</sup> Official and tentative methods of analysis of the Association of Official Agricultural Chemists. Washington. 1920. p. 94.

The reducing sugar, calculated as maltose before inversion was 5.51% on the basis of the moisture content of the bread (37.18%) and 5.82% after inversion. Such a slight increase in reducing sugar after inversion does not indicate that sucrose, if present, has contributed to the sweetness of the bread.

A comparison of the residual sugar content of bread made from straight doughs containing 2.5% of cane sugar and cerelose, respectively, (basis flour 100) as the only added sugar in the dough may also be mentioned. The total sugar content of the straight dough containing cane sugar was 2.00%, on the total weight of dough and the dough containing cerelose 1.85%, including the sugar present in the flour. The amount of yeast was 2% on the basis of flour as 100.

The formula of the two doughs was exactly the same, except one contained cane sugar and the other cerelose. They were mixed, fermented and baked under similar conditions on the same day, with but little less than an hour elapsing between the two mixings. The dough containing cerelose was mixed first, followed by the dough containing the cane sugar. The temperature of mixing was 80° F., first punch 81° F., second punch 82.5° F. for each dough. The total fermentation time was 190 minutes for each dough. They were then scaled, rounded, molded and panned by hand, proofed at 90° F. and then transferred to the Duhrkopf oven. The time of baking was 30 minutes at 475° F. The bread was cooled one hour, wrapped and sent to the laboratory.

The moisture content of the bread containing cane sugar was 37.25% and the bread containing cerelose 37.18%. The reducing sugar calculated as maltose was 2.97% before inversion and 3.30% after inversion in the bread from the dough to which cane sugar had been added. In the bread made from the dough containing cerelose, the reducing sugar before inversion calculated as maltose was 2.77% and after inversion 2.88%.

The slight increase in reducing sugar after inversion calculated as maltose noted in each of the breads does appear to indicate that sucrose (if the increase in reducing sugar is due to the inversion of this sugar) is present in amounts which would have a marked influence upon sweetness of taste. The taste of the bread made from the sponge and dough was somewhat sweeter than either the bread made with cane sugar or cerelose.

The residual sugars of bread are probably very largely composed of reducing sugars, among which maltose predominates.

Further work is in progress on the identification and determination of the residual sugars of bread made with increasing amounts of cane sugar, cerelose and other saccharine products, with especial reference to methods of analysis.

**COLLABORATIVE STUDY OF MOISTURE METHODS**

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(Received for publication July 8, 1925)

The two moisture methods that were studied by the Association of Official Agricultural Chemists during 1924 under the direction of Spencer,<sup>1</sup> were the vacuum or "umpire" method and the "routine" method. The dependability of the vacuum or "umpire" method is demonstrated beyond question. The inconveniences that attend the use of vacuum ovens, the expenditure of time, and the cost of complicated equipment led Spencer to seek a method that would give results comparable to those of careful vacuum drying and as a result the "routine" method was developed.

Spencer and his collaborators have shown that it is possible to obtain results by the routine method that agree very closely with those obtained by the vacuum method.

**Object of the Study**

Realizing the need of a rapid method for the determination of moisture in wheat flour, it was suggested that the collaborative moisture work of the American Association of Cereal Chemists be confined, for the present at least, to a study of the vacuum and routine methods, as proposed by Spencer.

"In this work it is proposed to study the vacuum or 'umpire' method and to apply such modifications as may be needful to learn the range of dependability of the 'routine' method when the temperature is varied while the other conditions remain constant."

**Methods Studied**

Method A (Proposed Standard Vacuum Method or "Umpire")

"Weigh accurately about two grams of the sample in a tared, covered dish. Loosen the cover and heat the dish and contents in a vacuum oven to 98-100° C. for five hours at a pressure of not more than 25 mm. (1 inch) of mercury. Tighten the cover on the dish and cool for 20 minutes in a desiccator. Weigh and calculate the loss in weight as moisture."

Method B (Proposed Routine Method)

"Weigh accurately about two grams of the sample in a tared, covered dish. Remove the cover and heat the dish and contents in air in an oven at 130° C. for one hour. Replace the cover on the dish

<sup>1</sup> Jour. Assn. Off. Agr. Chemists (1925). Vol. 8, pp. 301-311.



and cool in a desiccator for 20 minutes; weigh and calculate the loss in weight as moisture."

**Method C (Proposed Routine Method, 1st Modification)**

Heat at a temperature of 125° C. All other conditions same as in Method B.

**Method D (Proposed Routine Method, 2d Modification)**

Heat at a temperature of 135° C. All other conditions same as B.

**Conditions of Making the Tests**

1. Use samples of about 2 grams, accurately weighed in covered dishes.
2. Use aluminum dishes, cylindrical in shape, 18 mm. high and 60 mm. in diameter, with closely fitting covers.
3. After drying, cover the dishes tightly and place them in an efficient desiccator. Freshly ignited calcium oxide (lumps) or calcium carbide is recommended for a desiccating agent.
4. Collaborators were asked to make at least 4 determinations on each method studied.

One sample of spring wheat flour was sent to each collaborator. Considerable care was exercised in preparing the sample. First, the flour was very thoroly mixed by rotating at least fifty times on a large piece of smooth wrapping paper, after which it was immediately placed in ground glass-stoppered bottles and sealed with paraffin. The bottles were insulated against heat by wrapping in an abundance of tissue-paper before placing in mailing parcels.

**Discussion of Results**

Collaborative results obtained by method A, vacuum or "umpire," are given in Table I.

It will be noted that the maximum variation for all results given in the averaged column is 0.71%. If, however, we consider only the results that are strictly comparative, the variation is 0.23%. The latter variation falls between the maximum and minimum of results obtained by twelve of the sixteen collaborators. Results reported by collaborators 13 and 14 are higher by 0.134% and 0.504%, respectively, than the mean average of results reported by collaborators 1 to 12. Collaborators 15 and 16 failed to report the number of tests conducted.

Collaborative results obtained by method B, "routine," are given in Table II. Here it will be seen that the maximum variation in the averaged column is 0.46%, twenty-seven collaborators participating. It will be noted also (see Table V) that the mean of the averaged results obtained by method B is lower by 0.13% than the mean of the averaged results obtained by method A.

Table III gives results of twenty-five collaborators on method C, the maximum variation of individual averaged results being 0.58%. Collaborators 15 and 16 neglected to report the number of tests conducted.

Table IV represents results of collaborative study of method D. The maximum variation is 0.63%, twenty-eight collaborators participating. Several collaborators failed to follow directions as specified, which probably accounts for the wider variation of results exhibited by this method.

The figures in Table V represent the average results obtained by the several collaborators on the four methods. The results from methods B, C, and D show a fair degree of uniformity. However, if we eliminate from methods A and D several results that are not strictly comparative, as previously indicated, a striking uniformity in the results of all methods is shown. This is especially encouraging from the standpoint of the number of collaborators participating.

The means of all averaged results from each of the four methods studied are as follows:

Method A, Vacuum or "Umpire".....	13.38 %
Method B, "Routine," 130° C.; 1 hour.....	13.25 %
Method C, "Routine," 125° C.; 1 hour.....	13.218%
Method D, "Routine," 135° C.; 1 hour.....	13.306%

### Summary

1. The methods show a fairly close concordance in results. The results seem to indicate that "routine" method B and first modification C are slightly superior to "routine" modification D in this respect.

2. Drying approximately 2 grams of flour in vacuum at 98-100° C. at a pressure of not more than 25 mm. for a period of five hours, appears to give slightly higher results on the average than "routine" methods B, C, and D.

3. The data appear to be in accord with the results of previous collaborative work in that water is lost progressively with increased temperature when heated in air.

4. In the final interpretation, it appears from the data obtained that the "routine" method is dependable within a fairly wide range of temperatures.

5. The data obtained thus far seem to justify a further study of the "routine" method with the view of its adoption as a rapid and accurate method for the mill laboratory.

TABLE I

RESULTS OF MOISTURE DETERMINATIONS BY METHOD A, VACUUM OR "UMPIRE"  
Heated at temperature of 98° to 100° C.; time, 5 hrs.; pressure, 25 mm. or less

No. of collaborator	Type of vacuum oven used	No. of tests	Maximum	Minimum	Average
			Per cent	Per cent	Per cent
1	Freas	3	13.32	13.29	13.30
2	Freas	4	13.46	13.35	13.39
3	Freas	4	13.40	13.32	13.36
4	Freas	4	13.40	13.35	13.38
5	Freas	4	13.32	13.28	13.30
6	Freas	4	13.42	13.35	13.38
7	Freas	5	13.287	13.258	13.27
8	Freas	4	13.42	13.39	13.405
9	Freas	4	13.21	13.19	13.197
10	Special	4	13.22	13.17	13.19
11	Mojonnier	4	13.355	13.335	13.347
12	Mojonnier	4	13.44	13.38	13.42
13	Mojonnier	4	13.54	13.47	13.51
14	Freas	4	13.85	13.80	13.83
15	Mojonnier	*	.....	.....	13.12
16	Mojonnier	*	.....	.....	13.82

\*Number of tests not given.

TABLE II

RESULTS OF MOISTURE DETERMINATIONS BY METHOD B, "ROUTINE"  
Dried in air oven at temperature of 130° C. for 1 hour

No. of collaborator	No. of tests	Maximum	Minimum	Average	Variation†
		Per cent	Per cent	Per cent	Per cent
1	4	13.38	13.28	13.32	.10
2	4	13.41	13.30	13.37	.11
3	4	13.28	13.25	13.27	.03
4	4	13.13	13.09	13.105	.04
5	5	13.34	13.21	13.276	.13
6	4	13.10	12.95	13.013	.15
7	5	13.476	13.096	13.278	.38
8	4	13.28	13.22	13.255	.06
9	4	13.35	13.27	13.30	.08
10	4	13.37	13.26	13.312	.11
11	4	13.205	13.190	13.20	.015
12	4	13.42	13.37	13.39	.05
13	4	13.53	13.42	13.46	.11
14	4	13.45	13.30	13.40	.15
15	*	.....	.....	13.16	...
16	*	.....	.....	13.41	...
17	4	13.28	13.24	13.26	.04
18	4	13.19	13.17	13.177	.02
19	4	13.13	13.07	13.10	.06
20	4	13.26	12.98	13.09	.28
21	4	13.09	12.93	13.00	.16
22	3	13.34	13.33	13.335	.01
23	4	13.21	13.11	13.18	.10
24	4	13.36	13.22	13.29	.14
25	4	13.47‡	13.44‡	13.46‡	.03
26	4	13.28	13.17	13.22	.11
27	4	13.45‡	13.38‡	13.41‡	.07

\*Number of tests not given.

‡Dried in same oven with gluten puffs.

†Maximum variation for all average results—0.46%.

‡Calcium chloride used as a desiccating agent.

TABLE III

RESULTS OF MOISTURE DETERMINATIONS BY METHOD C, "ROUTINE," FIRST MODIFICATION  
Dried in air oven at temperature of 125° C. for 1 hour

No. of collaborator	No. of tests	Maximum	Minimum	Average	Variation†
		Per cent	Per cent	Per cent	Per cent
1	4	13.52	13.46	13.48	.06
2	4	13.36	13.30	13.33	.06
3	4	13.30	13.23	13.26	.07
4	4	13.09	13.03	13.055	.06
5	4	13.31	13.29	13.29	.02
6	4	13.165	13.03	13.112	.135
7	5	13.406	13.27	13.358	.136
8	4	13.18	13.11	13.157	.07
9	4	13.32	13.18	13.267	.14
10	4	13.47	13.27	13.36	.20
11	*	.....	.....	.....	...
12	4	13.38	13.33	13.36	.05
13	4	13.52	13.44	13.48	.08
14	4	13.40	13.35	13.375	.05
15	*	.....	.....	12.90	...
16	*	.....	.....	13.40	...
17	4	13.24	13.08	13.157	.16
18	4	13.19	13.06	13.142	.13
19	4	13.12	13.07	13.097	.05
20	4	13.11	12.87	12.98	.24
21	4	13.13	13.07	13.10	.06
22	4	13.335	13.130	13.201	.205
23	4	13.13	13.01	13.06	.12
24	2	13.24	13.23	13.235	.01
25	4	13.49	13.39	13.45	.10
26	3	12.93	12.87	12.903	.06

\*Number of tests not given.

†Maximum variation on all average results—0.58%.

TABLE IV

RESULTS OF MOISTURE DETERMINATIONS BY METHOD D, "ROUTINE," SECOND MODIFICATION  
Dried in air oven at temperature of 135° C. for 1 hour

No. of collaborator	No. of tests	Maximum	Minimum	Average	Variation
		Per cent	Per cent	Per cent	Per cent
1	3	13.39	13.36	13.37	.03
2	4	13.37	13.31	13.34	.06
3	4	13.36	13.32	13.34	.04
4	4	13.29	13.13	13.175	.16
5	4	13.41	13.35	13.38	.06
6	4	13.16	13.115	13.133	.045
7	5	13.486	13.34	13.43	.146
8	4	13.35	13.30	13.33	.05
9	4	13.41	13.28	13.345	.13
10	4	13.34	13.24	13.287	.10
11	4	13.26	13.19	13.228	.07
12	4	13.47	13.40	13.44	.07
13	4	13.69	13.60	13.63	.09
14	4	13.55	13.50	13.525	.05
15	*	.....	.....	13.18	...



TABLE IV—Continued

No. of collaborator	No. of tests	Maximum	Minimum	Average	Variation
		Per cent	Per cent	Per cent	Per cent
16	*	.....	.....	13.52	...
17	4	13.42	13.30	13.352	.12
18	4	13.25	13.16	13.20	.09
19	4	13.07	13.03	13.042	.04
20	4	13.24	13.04	13.14	.20
21	4	13.10	13.02	13.06	.08
22	4	13.385	13.29	13.339	.095
23	8	13.25	13.07	13.165	.18
24	3	13.35	13.18	13.27	.17
25	4	13.59†	13.50†	13.54†	.09
26	4	13.53	13.33	13.43	.20
27	*	.....	.....	.....	...
28	4	13.52	13.30	13.44	.22
29	*	.....	.....	13.00	...

\*Number of tests not given.

†Calcium chloride used as desiccating agent.

TABLE V

AVERAGE RESULTS OBTAINED BY THE SEVERAL COLLABORATORS ON THE FOUR METHODS

No. of collaborator	Method A, vacuum or "umpire"	Method B, "routine" 130° C.; 1 hour	"Routine" 1st modification C, 125° C.; 1 hour	"Routine" 2d modification D, 135° C.; 1 hour
1	13.30	13.32	13.48	13.37
2	13.39	13.37	13.33	13.34
3	13.36	13.27	13.26	13.34
4	13.38	13.105	13.055	13.175
5	13.30	13.276	13.29	13.38
6	13.38	13.013	13.112	13.133
7	13.2706	13.278	13.358	13.43
8	13.405	13.255	13.157	13.33
9	13.197	13.30	13.267	13.345
10	13.19	13.312	13.36	13.287
11	13.347	13.20	.....	13.228
12	13.42	13.39	13.36	13.44
13	13.51	13.46	13.48	13.63
14	13.83	13.40	13.375	13.525
15	13.12	13.16	12.90	13.18
16	13.82	13.41	13.40	13.52
17	.....	13.26	13.157	13.352
18	.....	13.177	13.142	13.20
19	.....	13.10	13.097	13.042
20	.....	13.09	12.98	13.14
21	.....	13.00	13.10	13.06
22	.....	13.335	13.201	13.339
23	.....	13.18	13.06	13.165
24	.....	13.29	13.235	13.27
25	.....	13.46	13.45	13.54
26	.....	13.22	12.903	13.43
27	.....	13.41	.....	.....
28	.....	.....	.....	13.44
29	.....	.....	.....	13.00
Average	13.38	13.25	13.218	13.306